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CONVENTIONAL AND ADVANCED ALUMINUM ALLOYS
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# A Study of Environmental Characterization of Conventional and Advanced Aluminum Alloys for Selection and Design

Phase I - Literature Review

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A STUDY OF ENVIRONMENTAL CHARACTERIZATION OF CONVENTIONAL AND AVANCED ALUMINUM ALLOYS FOR SELECTION AND DESIGN

PHASE I - LITERATURE REVIEW

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#### FOREWORD

The problems arising with proliferating stress corrosion cracking (SCC) test methods and a need to relate various types of laboratory test results with each other and with service requirements has long been recognized. Technical direction at Alcoa Laboratories identified the situation as an industrial problem, and this contracted effort was conceived (funded by NASA Langley with W. B. Lisagor, monitor) with the objective of clarifying relationships between various SCC testing techniques and providing guidance on optimum characterization methodlogy for aluminum alloys. The program was constructed in two phases. The first, Phase I, was to be a review of the literature relating to: (a) the SCC performance of high strength aluminum alloys, and (b) comparison of SCC characterization by different methods. A prime objective of this survey was to aid in formulating an experimental program, to be done in Phase II of this contract, with the objective of determining the type or combination of accelerated SCC test procedures most suitable for selection and design of high strength aluminum alloys.

This report contains four technical progress reports submitted in partial fulfillment of the contracted Phase I literature review. These reports are close reproductions of the original technical progress letters submitted during the contracted time frame. Each report, or section, has its own conclusions or summary of salient observations. A few major impressions of the findings in the literature and the present state-of-the-art of SCC testing aluminum

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alloys are presented in Part V of this report. D. O. Sprowls performed the literature review and is the principal author of this report. Section III, on Mechanical Aspects of SCC Testing was conauthored by D. O. Sprowls, R. J. Bucci and R. L. Brazill. The final report was edited by R. J. Bucci and D. O. Sprowls. Review of the manuscript by J. D. Walsh is gratefully acknowledged.

In a separate report covering the contracted Phase II effort, an updated summary of the reviewed literature is presented, with greater emphasis given to the mechanical aspects of SCC testing. Of particular interest in the Phase II overview are sections on materials selection, problems with state-of-the-art accelerated test methods, and introduction to a new approach to smooth specimen testing, "the breaking load method," which is viewed to hold considerable promise as a much improved quantitative approach for assessing SCC behavior. Considering input from all of the above, the Phase II experimental program proceeded with the objective of advancing the breaking load method and to verify the claimed advantages of this approach over current state-of-the-art SCC characterization procedures. The results of this investigation are presented in the Phase II final report, which was coauthored by D. O. Sprowls, R. J. Bucci, B. M. Ponchel, R. L. Brazill and P. E. Bretz.

Ву

D. O. Sprowls

First Technical Progress Report Submitted in Partial Fulfillment of:

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A STUDY OF ENVIRONMENTAL CHARACTERIZATION OF CONVENTIONAL AND ADVANCED ALUMINUM ALLOYS FOR SELECTION AND DESIGN

PHASE I Review of the Literature

Reported For: Contract Period Ending April 16, 1981

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In this review of literature on stress-corrosion cracking (SCC) of aluminum alloys, emphasis is being placed on newer test techniques and advanced aluminum alloys.

During the 1960's two new test techniques involving different mechanical factors emerged: one involving mechanically precracked test pieces and the second involving constant strain rate tests. Prior to 1965, the assessment of SCC was done with constant load or constant strain tests of smooth and notched test pieces. The impact of these new methods on SCC characterization is covered in References 1-6.

The standardization of stress corrosion testing methods in the U.S.A. was started in 1964 when ASTM Committee G-1 was formed for the Corrosion of Metals, with subcommittee G01.06 on Stress-Corrosion Cracking and Fatigue. In the 1980 Annual Book of ASTM Standards there are now twelve standards on stress corrosion testing, eight of which are applicable to the characterization of aluminum alloys (G30, G38, G39, G44, G47, G49, G58, and G64) (Ref. 7). These involve testing wih various types of constant strain or constant load tests of smooth specimens. The most recent of these standards (G64-80), which is a Standard Classification of the Resistance to Stress-Corrosion Cracking of High Strength Aluminum Alloys, is based on service experience, if available, or on laboratory tests of standard smooth specimens at specified stress levels. With regard to other test methods, a statement from that standard is quoted: "5.2 Other types of tests using precracked specimens or dynamic laoding have promise as

alternative or supplementary methods, but they presently require better understanding and standardization." (This is still true at the time of this writing, August 31, 1984).

Specific sections of ASTM Subcommittee G01.06 have been organized for the purpose of developing standard procedures for the use of precracked specimens (Section 4) and dynamic testing (Section 5). Work in this direction, including round robin testing, is in progress.

The ASTM is also involved with the International Standards Organization (ISO/TC 156) on Corrosion of Metals and Alloys through WG2 on Stress Corrosion Cracking. Several ASTM documents are under consideration for acceptance as ISO standards.

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II. AVOIDING STRESS CORROSION (SCC) CRACKING IN HIGH STRENGTH ALUMINUM ALLOY STRUCTURES

Ву

D. O. Sprowls

Second Technical Progress Report Submitted in Partial Fulfillment of:

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A STUDY OF ENVIRONMENTAL CHARACTERIZATION OF CONVENTIONAL AND ADVANCED ALUMINUM ALLOYS FOR SELECTION AND DESIGN

PHASE I Review of the Literature

Reported For:

Contract Period Ending September 16, 1981

#### A. NECESSARY CONDITIONS FOR SCC

Stress corrosion cracking (SCC) is a time dependent process that involves the interaction of sustained tensile stress and a corrodent at the surface of a metallurgically susceptible material (Figure 1). All four of the necessary conditions must be simultaneously present and the process is synergistic; i.e., the damage due to SCC is greater than the additive effects of the individual conditions. SCC is recognized as a potential problem with various alloys and tempers of all structural metals (1, 2).

With aluminum, metallurgical susceptibility is confined to the higher strength alloys in certain tempers. The cracking proceeds along an intergranular path\* until the strength of the part is reduced to the point where fracture may occur. Because thick sections of most high strength aluminum alloy wrought products have a directional grain structure, the resistance to SCC of a susceptible alloy and temper also is influenced by the direction of stressing relative to the macrostructure (3-7). This is illustrated for 7075-T651 rolled plate in Figure 2.

SCC in service can result in premature failure because it usually occurs at nominal stress levels far below the

<sup>\*</sup> While transgranular SCC has been recognized under certain conditions in laboratory tests, this mode has not been identified in the historical cases of SCC in service (8).

engineering yield strength of the product. Unlike (corrosion) fatigue cracking which only propagates during cyclic operating loads, SCC can continue under the driving force of sustained residual tensile stress. Theoretically the time to failure by SCC can be lengthened, or the cracking even prevented by lowering the tensile stress; but for a highly susceptible material, loaded in the short transverse direction, the stress must be reduced to impractically low values. The interaction between sustained tensile stress and the metallurgical susceptibility of the material is illustrated in Figure 3 by the comparative performance of different tempers of 7075 alloy plate (8).

SCC will not occur in a vacuum or in a dry atmosphere (less than about 0.1 per cent relative humidity (5). SCC can occur in ordinary environments with water being the essential ingredient, present as vapor in the atmosphere and as liquid in aqueous or organic solutions. Both the initiation and propagation of SCC in natural environments are accelerated by increase in moisture, temperature, chlorides (traces of which are present almost everywhere) and various industrial contaminants. Further discussion of environmental factors is given in Section IV.

In estimating the probability of an SCC problem in a structural component, it is necessary to consider the following (9):

(a) The material -

Alloy
Temper
Product form (plate, extrusion, forging, ...)
Section size (thickness)

(b) Magnitude of sustained tensile stress -

Residual stress at the surface of finish machined part Fabrication and assembly stresses Service stresses (design)

- (c) Direction of sustained tensile stress relative to grain orientation in the component.
- (d) Geometry of the component and stress profile through the thickness.
- (e) Nature of the environment and degree of surface protection.

#### B. SCC SERVICE FAILURES

Most instances of SCC failures of aluminum alloys in service are found in aircraft components where high strength alloys are used extensively. Prior to World War II, the mill product forms and construction methods used in metal aircraft seldom created potential SCC conditions. Pressed-in bushings represented about the only components occurring frequently in normal design that required attention with respect to stress corrosion. Therefore, SCC occurred infrequently and, for the most part, designers and builders of high strength aluminum structures were not accustomed to preventing it. The few SCC occurrences were diagnosed and the problems solved with little fanfare (10-12).

After World War II (during the 1940's - '60's), increasing numbers of stress corrosion problems appeared in aerospace vehicles (13-17). The following material was excerpted from an extensive survey of failure reports summarized by Speidel (17): "We have plotted (Figure 4) the estimated total number of stress corrosion service failures which occurred with aerospace products in Western Europe and North America vs. the year in which these failures were reported. The data ... have been extracted from over three thousand individual failure reports from six aerospace companies and a number of government agencies and research laboratories in the U.S. and five countries in Western Europe. The SCC service failures have been observed with such items as small aircraft (the majority of failures) helicopters, jet aircraft, and rockets. Only significant failures are reported (e.g., SCC of a big forging is one failure, SCC of ten identical bolts is also listed as one failure). The majority of all reported failures of most fleets was included in the statistic but a certain amount of extrapolation was necessary to estimate the total number of failures, since it is impossible to get all failure reports. Moreover, all classified information is excluded, and of course all failures that were never reported. Apart from that, the order of magnitude, the trend and the relative number of failures for the various alloy systems is considered to be correct and the following conclusions can be drawn from Figure 4:

"The number of stress corrosion service problems rose from 1960 until 1968, and it may be interesting to speculate on the reasons that caused a reversal of this trend in 1969 and 1970.

Among the possibilities:

- (1) designers have learned from their failures and introduced less susceptible alloys, better surface protection, inhibited environments, and reduced sustained stresses;
- (2) research and development efforts have paid off and provided better alloys as well as a greater awareness of the problem;
- (3) the declining market situation in the aerospace industry has resulted in less aerospace products and thus less failures."

"It should also be emphasized that in 1970 the number of stress corrosion service failures with high strength alloys was greater than in 1965."

"In addition, it is important to point out that the service failures listed (Figure 4) almost never resulted in crashes or other catastrophic vehicle failures. The vast majority of the SCC failures was of structural parts which were routinely replaced during inspction and maintenance. However, the failures resulted in significant economic losses, due to the cost of replacement and (often more significant) due to the cost of down time."

#### 1. "Specific Causes for SCC Service Failures:

"A more detailed analysis of the thousands of failure reports upon which Figure 4 is based, has provided the following information:

#### 2. "Materials:

"Alloys 7079-T6, 7075-T6, and 2024-T3 contributed to more than 90 percent of the service failures of all high-strength aluminum alloys.

#### 3. "SCC Crack Initiation Sites:

"The distribution of known initiation sites is given in Table 1. The variety of sites and causes for SCC crack initiation makes it appear a hopeless task to fully protect against SCC by surface treating a structure which is built with inherently susceptible materials. This points out the necessity to reduce the growth rate of stress corrosion cracks to a level low enough that cracks do not become critical during the lifetime of a structure.

# 4. "Sources of Stresses Causing Stress Corrosion Crack Propagation:

"These are listed in Table 2. Obviously, residual stresses from heat treatment and fabrication are by far more frequent causes of SCC than service stresses. This is partly because most SCC failures occurred with large forgings where residual stresses are unavoidable."

"The reason why actual stress corrosion service failures with high-strength aluminum alloys occur almost exclusively on parts with thick sections is the well known combination of grain flow during processing and the direction of residual and applied load in service. During forging the grains are flattened in the parting plane, and during rolling the grains are flattened in the plane of the plate. The resulting highly directional grain shape is not changed during subequent heat treatment, because minor constituents such as 2r, Cr, and Mn form small particles of intermetallic phases which prevent the large-angle grain boundaries from moving during solution heat treatments. In thin gage material like sheet, there is normally no stress applied in the short transverse section (i.e., in the direction perpendicular to the plane of the sheet). Thus, intergranular SCC cracks cannot grow easily along the grain boundaries, the majority of which are in the plane of the sheet. In thick sections, on the contrary, significant residual and service stresses can exist perpendicular to the preferred plane of the grain boundaries, which explains why stress corrosion cracks are mostly observed in thick sections."

Specific examples of typical service failures are illustrated in Figures 5-10. The spool shown in Figure 5 is from a fishing reel used for salt water fishing. The use of alloy 2024-T4 was not intended; when the cracked reel was received for examination it was

reported to be 6061-T6. If the rod stock had been 6061-T6 (SCC resistant alloy) or 2024-T351 (stress relieved); this failure would not have occurred. Another example of a failure from similar cause is shown in Figure 6. Figure 7 illustrates a failure caused by residual stresses produced by fabrication and procedures to avoid the problem. Fortunately in this instance the potential service failure was discovered by a laboratory simulation test of the intended procedure. Examples of typical failures caused by installation stresses are sketched in Figures 8 and 9. In the latter cases, it is also shown how short transverse stresses can be developed in unexpected situations when thin parts are machined out of relatively thick parts. Residual stresses produced in welded assemblies also have caused SCC as shown in Figure 10 along with procedures for avoiding failure.

The greatest danger arises when residual, assembly, and service stresses combine to produce high sustained tensile stress at the metal surface (18). Theoretically, one way to avoid SCC of susceptible material is to control the stress at a safe level. Several approaches are available through proper design, fabrication and assembly practices. It is good practice to pay close attention to all of these, even when alloys with improved resistance to SCC are used.

#### C. SERVICE STRESSES (DESIGN STRESSES)

Design stresses have not caused SCC in aluminum alloy structures, except in assemblies where the part is held under high tensile load on an "around-the-clock" basis. Examples involve interference fit bushings and fasteners, clamps, and hydraulic fittings. Care must be exercised when products with appreciable susceptibility to SCC are used in such components. Stresses from these sources are different from other installation stresses in that control of the applied stress is practicable.

In general, the designer of high strength structures in aluminum has no problem with regard to SCC as far as the external design loads are concerned, because the primary loading is usually in the highly resistant longitudinal or long transverse grain orientation. Moreover, other design criteria, especially the fatigue requirements, generally will ensure that the operational stresses will not be high enough to promote SCC failure. Most fatigue loads for which structures are designed are of relatively short duration and do not contribute significantly to SCC. However, there are some kinds of components, such as hydraulic cyclinders, which must endure cyclic loading superimposed on a sustained load. In such situations, the contribution of the intermittent loading must be taken into

consideration. Laboratory tests of hydraulic cyclinders have shown that SCC may occur under cyclic loading at low frequencies, with both SCC and corrosion fatigue interacting to produce failures in shorter times and fewer cycles than for either phenomenon alone (19).

It is conceivable that as fabrication practices are improved to minimize residual stresses in structures, then the operational stresses may become of increased concern. If a "threshold stress" above which failure will occur by SCC can be identified for a given component, then this must be considered among other design criteria.

#### D. PRESENT DESIGN PHILOSOPHIES FOR AVOIDING SCC

Two basic design concepts which found original application as safeguards against metal fatigue also are applicable to SCC failures. A brief comparison of these two different approaches is given here because the design concept to be applied to a particular structure can influence the strategy for avoiding SCC (20).

#### l. Safe Life

The safe-life concept is the one most often considered applicable to avoiding SCC problems in high strength aluminum alloy structures. Several observations derived from service experience have contributed to this approach:

(a) SCC in service generally has resulted from residual and assembly stresses (non-design) acting in the short transverse grain orientation, and may propagate in directions unrelated to the service loads; (b) the magnitude of such unplanned stresses is generally unknown, and the crack tip stress intensity factor usually changes as the crack extends; it can either increase or decrease, depending upon the type of loading; (c) SCC propagation rates in specific components are far less predictable than in the case of fatigue; (d) materials that have been involved in service SCC problems are capable of developing relatively high SCC propagation rates.

Traditionally, few attempts have been made to separate component failure into initiation and propagation stages. Discovery of SCC in a part, regardless of whether unstable fracture has occurred, usually has led to the retirement of that part. Thus, in the safe-life design concept, based on the premise that the total life of a part consists primarily on the initiation of a visible crack, the strategy is to prevent cracks from forming. The design and materials selection rely heavily on closely related service experience and comparison of accelerated coupon-type test results, usually of statically loaded smooth specimens.

#### 2. <u>Damage Tolerance</u>

For fracture control in high-performance aircarft, increasing use is being made of the damage tolerant approach by which design concepts may be qualified as either "slow crack growth" or "fail-safe"structures (21). Initial flaws are assumed to exist as a result of manufacturing and processing operations. Given a crack-like flaw corresponding to the maximum size escaping reliable detection, life of the part is assumed to be spent propagating this flaw to the critical size which results in fracture. The damage tolerance evaluation of a structure is intended to ensure that, should cracking occur, the remaining structure can function until the damage is detected and remedied. The general design strategy, therefore, is to select materials, configurations, and stress levels that provide a slow rate of crack propagation while maintaining high residual strength.

The Damage Tolerant Design Handbook, however, presently recommends, that, "the best design policy for handling SCC is to prevent it, rather than controlling its growth as done for fatigue cracking" (22).

#### 3. Allowable Stress Level

An SCC "threshold stress" is frequently sought as a useful characterization parameter for an engineering material, i.e., the stress level below which SCC will not be anticipated. The "threshold stress" or "threshold stress intensity factor" for SCC is not an absolute property as is often implied. Although there may be an apparent threshold level of stress for the initiation of SCC - as suggested by various smooth specimen data (Figure 2) - any SCC threshold determined in the laboratory is test dependent and must be identified with the controlling conditions such as environment, length of exposure, size of test specimen, method of loading, etc. Moreover, it must be described in terms of a specified probability of failure (low) and confidence level (high).

It is not advisable [Brown calls it "certain folly" (24)] to design for a sustained tensile stress just beneath a threshold stress measured in an accelerated test no matter how carefully that determination may have been made. A sizable margin must be allowed because unexpected stresses from heat treatment, fitup, thermal expansion, and local stress concentrators usually are present, and one does not

**(+)** 

want to run the risk that unknown stresses from such causes may elevate the effective stress above the anticipated SCC threshold. Moreover, in the presence of certain crack-like flaws, stress corrosion cracks may grow at stresses lower than the apparent threshold stress developed from smooth specimen results.

#### E. CONCLUSIONS

Analyses of service failures of various types are needed in terms of actual requirements of the application. These should be considered in relationship to the relative importance of initiation and propagation of SCC, the type of loading and the design philosophy used for the structure. Learning from such experience can be expected to impact on the strategy for avoiding SCC and the test methodology for material selection and design.

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# TABLE 1. INITIATION SITES OF STRESS CORROSION CRACKS IN HIGH STRENGTH ALUMINUM ALLOYS (FROM SPEIDEL REF. 17)

Stress raisers due to design	
(bore hole, sharp radius, etc.)	25 pct
Holes for interference fit pushings	15 pct
Corrosion pits	12 pct
Fatigue cracks	5 pct
Galling, fretting, wear	5 pct
Intergranular corrosion, exfoliation	4 pct
Not known	34 pct

# TABLE 2. SOURCES OF STRESSES CAUSING PROPAGATION OF STRESS CORROSION CRACKS IN HIGH STRENGTH ALUMINUM ALLOYS (FROM SPEIDEL REF. 17)

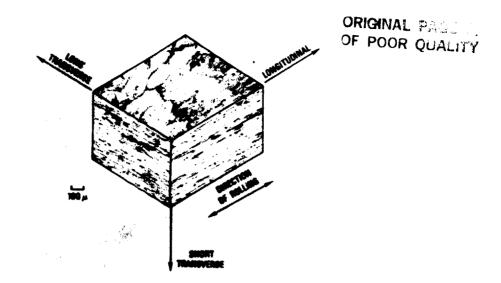
Residual stress (from heat treatment and fabrication) Installation stresses (fit-up stresses, improper	40 pct
shimming, torque)	25 pct
Service stresses (amplified due to stress raisers) Not known	25 pct
	10 pct

STRESS CORROSION CRACKING

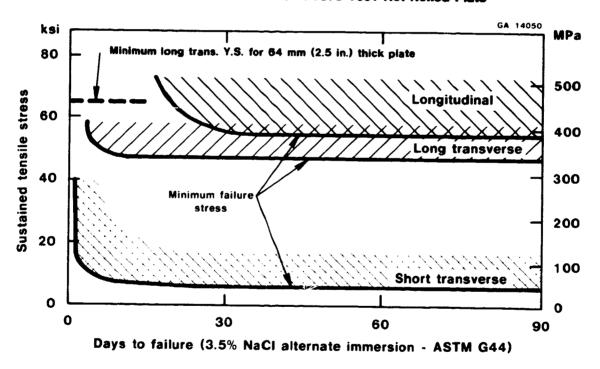
Environment

Environment

Necessary Conditions for Stress-Corrosion Cracking Figure 1



Directional Grain Structure of 7075-T651 Hot Rolled Plate

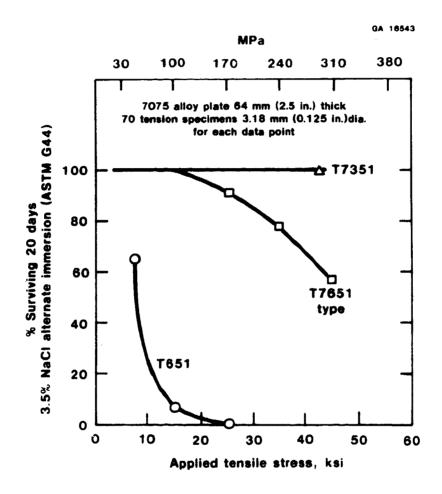


Tests were made on 3.18 mm (0.125 in.) diameter tension specimens machined from the mid-plane of 7075-T651 plates of various thicknesses. The solid line, lower bound defines the SCC performance of test specimens with different orientation to the grain structure. Note the relatively low stress levels at which short transverse specimens failed compared to the long transverse and longitudinal specimens (Ref. 9).

Effects of the Magnitude of Sustained Tensile Stress and Its Orientation Relative to the Grain Structure on the SCC Resistance of a Metallurgically Susceptible Material

Figure 2

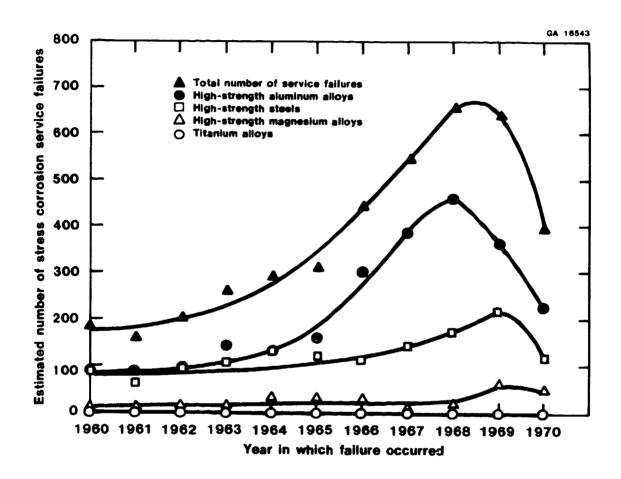
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The metallurgical susceptibility to SCC is significantly less for the T7351 and T7651-type tempers. Their improved performance compared to the T651 is indicated by the higher percent survival curves shown as a function of stress (Ref. 8).

Effect of Temper on SCC Performance of Alloy 7075 Plate Stressed in the Critical Short Transverse Direction.

Figure 3

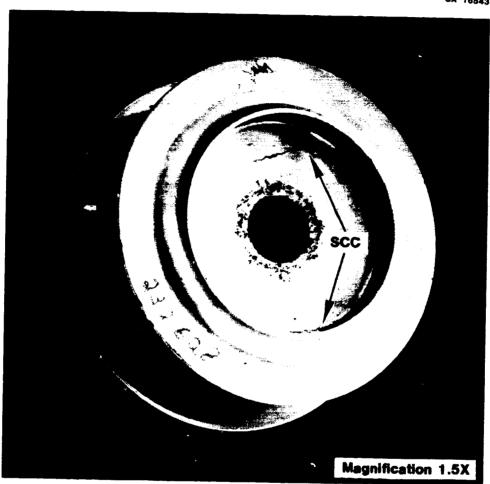


Estimated Number of Stress Corrosion Service Failures of Aerospace Products in Western Europe and North America from 1960 to 1970 (from Speidel Ref. 17).

Figure 4

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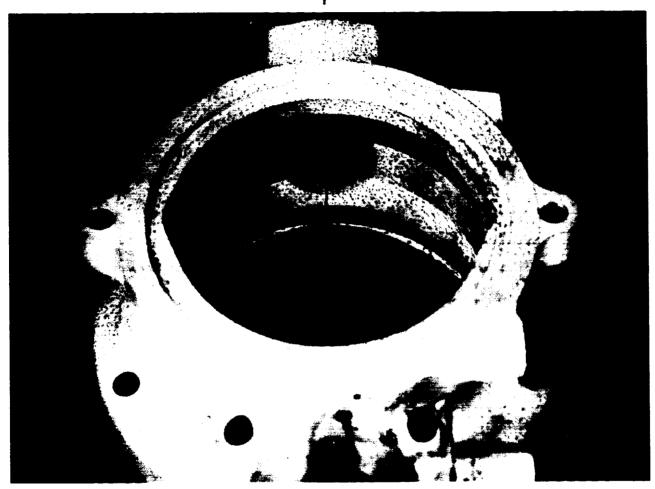
Stress-corrosion cracks in the flanges of a spool machined from 2024-T4 rod stock. The removal of large amounts of stock resulted in transverse residual tensile stress on the machined surface. This problem would have been avoided by selection of the stress relieved T351 temper (Ref. 9).

Example of SCC Caused by Residual Stresses from Quenching Figure 5

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Parting plane

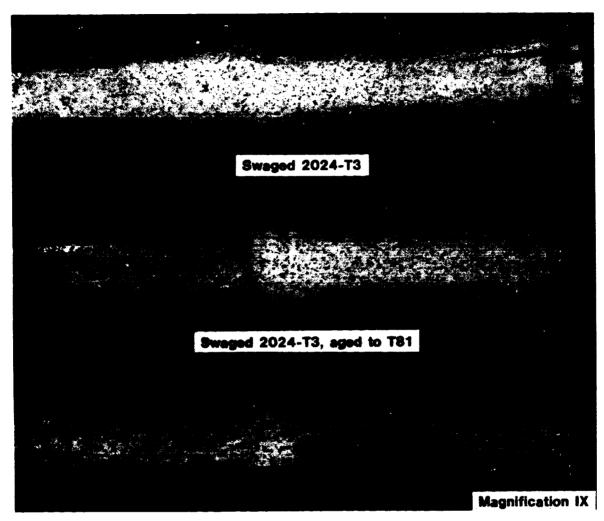
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Die forged valve body of 7075-T6 alloy showing SCC at the intersection of the machined hollow boss with the main chamber. This crack is parallel to the metal flow lines of the parting plane and, hence, is a short transverse failure.

Example of SCC Caused by Residual Stresses from Quenching Figure 6

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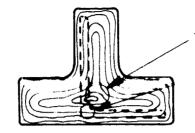
Swaged 2024-0, H.T. to T42

Tubes with swaged ends (20% reduction) of various tempers of 2024 alloy exposed to 3-1/2% NaCl solution by alternate immersion for 84 days. SCC can be avoided simply by using the proper sequence of swaging and tempering. Corrosion products chemically removed after exposure (Ref. 9).

Example of SCC in a 2024-T3 Tube Caused by Residual Stresses from Fabrication Figure 7

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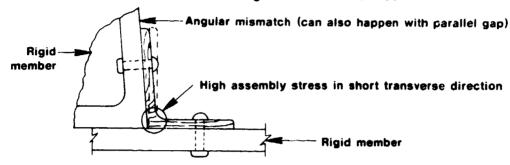
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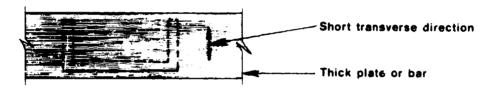
Transverse direction

Short transverse direction—vertical Long transverse direction—horizontal

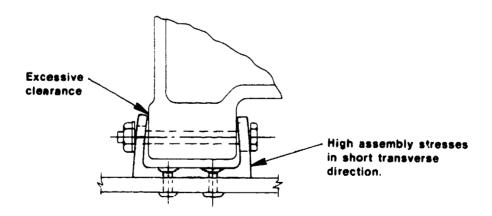
Location of machined angle with respect to transverse grain flow in thick tee



(A)-Locked in assembly stresses from mismatch



Location of machined channel in plate or bar

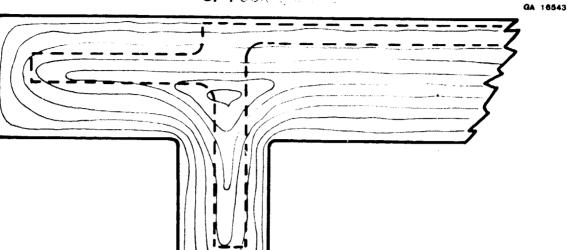


(B)-Locked in assembly stresses from excessive clearance

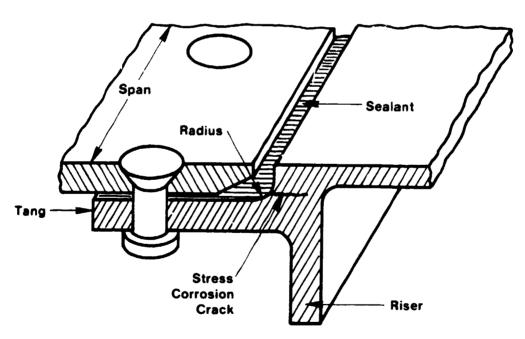
Examples of Short Transverse Tensile Stresses Developed During Assembly of Thin Sections Machined from Thick Products (Ref. 9). See Also Figure 3.

Figure 8

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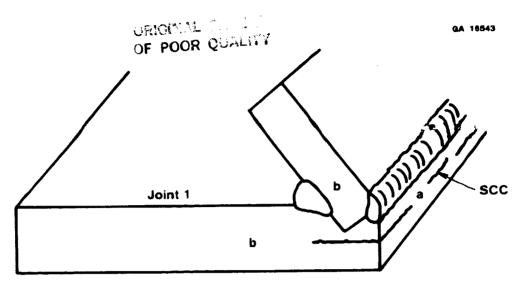


Location of machined wing plank with respect to transverse grain flow in thicker extruded section



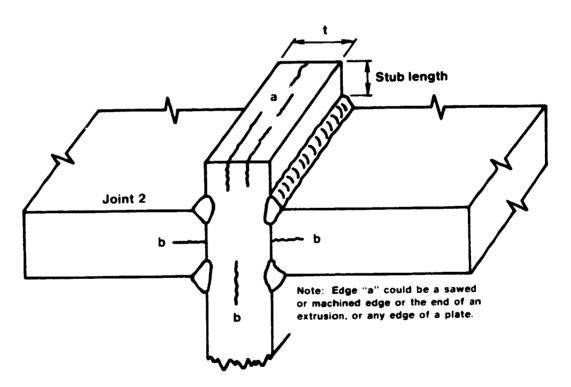
The crack was caused by short transverse stress imposed by assembly mis-match and corrosion products resulting from exfoliation corrosion of the faying surfaces where moisture had gained entrance to the gap between the planks (Ref. 14).

Diagram of a Typical Wing Plank Joint Showing the Location of SCC in the Tang Radius
Figure 9



#### Recommended:

- "Butter" with overlay of weld metal, or shot peen edge "a"
- "Butter" terminal edge "b" in vicinity of welds



#### Recommended:

- Make stub length at least 1.5t, or
- "Butter" with overlay of weld metal, or shot peen edge "a"
- "Butter" terminal edges "b" in vicinity of welds

Methods of Avoiding SCC Caused by Residual Welding Stresses Acting in the Short Transverse Direction Across Exposed Edges (Ref. 9).

Figure 10

# III. MECHANICAL ASPECTS OF STRESS CORROSION TESTING FOR ALLOY DEVELOPMENT AND SELECTION

ВУ

D. O. Sprowls, R. J. Bucci, and R. L. Brazill

Third Technical Progress Report Submitted in Partial Fulfillment of:

NASA CONTRACT NAS1-16424

A STUDY OF ENVIRONMENTAL CHARACTERIZATION OF CONVENTIONAL AND ADVANCED ALUMINUM ALLOYS FOR SELECTION AND DESIGN

PHASE I REVIEW OF THE LITERATURE

Reported For:

Contract Period Ending November 16, 1981



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#### A. INTRODUCTION

Accelerated stress-corrosion cracking (SCC) tests are a vital part of materials evaluation for alloy development, selection, and design. Over the years many testing techniques have been developed to measure material response to agressive environments under stress. It is questionable whether any single test method can provide all of the information desired for a given service application. Also, the material behavior observed in a laboratory accelerated test may not be the same as that occurring in service over a long exposure period. This report reviews the mechanical aspects of SCC test techniques and weighs the advantages of each with the goal of determining some optimum techniques for characterizing SCC behavior in accelerated tests. The environmental aspects of SCC testing are discussed in Section IV.

A stress corrosion failure can be considered to occur in sequential stages, although it may not be possible to clearly separate the stages (1). First is the relatively slow initiation (incubation) stage in which corrosion reactions take place but which do not affect the mechanical properties of the part (specimen). This is followed by the formation of localized sites of corrosion attack (fissures) which create stress concentrations and the establishment of a small number of "well defined" cracks. Stable subcritical growth (propagation) of one of these cracks to a critical size results in mechanical fracture. Thus, the stress corrosion life of a part or test specimen is the sum of the initiation and propagation lifetimes,

as shown schematically in Figure 1. Complete fracture may not be involved in the SCC failure of some parts or specimens, depending on the method of loading and the criterion of failure (as for example, a cracked and leaking hydraulic line fitting). A considerable proportion of the time required for the occurrence of an SCC failure can be involved in the initiation stage, as shown by the specimen-life curves in Figures 2 and 3. In other cases, such as constant deformation loaded thick sections, the propagation time can become dominant.

In general, three approaches are used for SCC testing and evaluation. The older, traditional approach involves statically loaded smooth specimens in which both the successive stages of SCC initiation and propagation occur in the usual manner. A mechanically accelerated technique introduced in recent years involves dynamic loading at a constant rate of strain instead of static loading. The rate of strain increase can alter the initiation and propagation lifetimes; hence it is not clear whether initiation or propagation is the dominant test response. The third testing approach involves mechanical acceleration by introducing a flaw (crack) in the specimen prior to environmental exposure. With this technique only the propagation stage is considered. This conservative approach is based on the premise that stress concentrators generally are present in engineering structures when they are put into service, and the most significant part of the stress corrosion life is the propagation stage. The following discussion gives a more detailed description of the various SCC testing methods and their relative merits.

#### SMOOTH SPECIMEN TESTS

в.

#### 1. Static Loading

The traditional method of measuring SCC susceptibility in the laboratory is by exposing smooth specimens to agressive environments while stressed by application of a constant load or constant strain. The true net section stress, however, increases as the depth of the corrosion fissures increase. This effect varies with testing conditions, such as type of loading and distribution of corrosion cracks, as indicated in Figure 4 (4, 5).

The usual testing procedure is to expose to the corrosive environment, several sets of replicate specimens loaded to various fractions of the material yield strength. The time to failure is determined as a function of the applied stress.

Also, the probability of failure at a particular exposure stress can be determined as a function of exposure time. Examples of such data for 7075 alloy plate are given in Figures 5 and 6.

Note in Figure 5 that specimen orientation relative to plate rolling direction has a significant effect on the performance of alloy 7075-T6. For each orientation there appeared to be a minimum stress below which the specimens were not likely to fail (threshold stress). It is apparent from Figure 6, however, that determination of a threshold stress can be influenced by a number of factors, such as specimen type and size and length of exposure.

4

A variety of smooth specimen types are used in SCC tests, depending on the product, thickness, and end use. The most widely used specimens are tension test coupons (ASTM G-49), C-Rings (ASTM G38), Bent Beams (ASTM G39), and U-Bends (ASTM G30). The use of these specimens for SCC tests has been standardized to facilitate test comparisons (8). Direct tension specimens are simple to test but cannot be used for short-transverse SCC testing of products less than about 37 mm (1.5 in.) thick. C-ring specimens can be used for short transverse tests of section thicknesses as low as 19 mm (0.75 in.). It should be recognized, however, that the SCC lifetime of a given material can be influenced by the type and size of specimen (Figure 6) and the method of loading (Figure 7). Thus, a threshold stress for SCC is not a material property, and any threshold estimates should be qualified with regard to the test conditions and the significance level. Such test results, however, do provide a useful means of ranking material, as in the ASTM standard for classifying the resistance to SCC of high strength aluminum alloys, G64-80 (Ref. 8).

#### 2. Dynamic Loading (Constant Extension Rate)

The constant strain rate test is a method for mechanically accelerating the assessment of susceptibility to SCC. This technique consists of straining specimens at rates in the range from  $10^{-8}$  to  $10^{-4}$  (in./in)/sec. under controlled environmental conditions. Susceptibility to SCC can be quantified in terms of various parameters such as maximum load, energy to fracture, and reduction in area of elongation (10).

**(4)** 

Generally, a critical strain rate exists for a particular system where a maximum stress corrosion effect is observed. At this critical strain rate there is a balance at the crack tip between deformation, dissolution, film formation and diffusion to maximize the SCC effect. At very fast strain rates, ductile fracture takes place before the necessary corrosion reactions can occur. With strain rates that are very slow, it may be possible for film repair to take place and reduce the detrimental effect of SCC reactions (11, 12). The general behavior of stress corrosion susceptibility with strain rate is shown in Figure 8. It has been proposed by Parkins (13) that "... stress or stress intensity, per se, may be less important than the strain rates they produce."

Work done on aluminum alloys by the slow strain rate method has been confined largely to comparing various alloys in a given environment and to determining the effect of loading rate on the SCC susceptibility of these materials. Most of the results available in the literature, however, cannot be effectively analyzed to determine whether or not susceptibilities indicated by this technique are consistent with known effects of certain metallurgical treatments. Limited tests by Maitra (14) showed that slow strain rate tests of incrementally aged 2124-T351 alloy plate (increased aging known to increase resistance to SCC) were in agreement with conventional test results (Figure 9). Loss in fracture energy and ductility compared to that in air were the most sensitive indicators of changes in resistance to SCC with artificial aging.

Although the constant strain rate technique shows promise as a means of ranking environments and possibly alloys, additional experience is needed to optimize loading rates and criteria of susceptibility. Moreover it is not clear how the test results relate to service needs (15).

#### C. PRECRACKED SPECIMEN TESTS

### 1. Application of Linear Elastic Fracture Mechanics (LEFM)

The third approach to assessing SCC susceptibility is to use fracture mechanics type specimens containing an established flaw, usually a crack formed by fatigue or tensile "pop-in" (16, 17). The purpose of the precrack is to ensure the initiation of SCC at a site, i.e., the crack tip, where the LEFM relationships are valid. Thus, fracture mechanics is applicable only to the propagation of SCC, as it is assumed that crack nucleii already have formed. The primary objective in performing SCC tests by this method is to determine the threshold stress intensity factor below which SCC will not propagate. Another objective is to determine the rate of SCC propagation, da/dt, as a function of the mechanical driving force,  $K_{T}$ , under controlled conditions. It is generally accepted that in order to fully characterize the resistance to SCC by this method, it is preferable to obtain the complete curve of  $K_{T}$  vs. da/dt (18). The advantages of this approach are twofold. First, only one stage (propagation) of stress corrosion is considered, thus hopefully eliminating the combined action of all prior stages as a measurement variable. Second, the aplication of linear elastic fracture mechanics (LEFM) to cracked bodies allows the stresses and strains near the crack tip process zone to be determined so long as the SCC occurs below gross yielding (19). From this information the mechanical driving force for cracks in many configurations can be quantified in terms of the stress intensity factor, K, which describes the magnitude of the elastic stress-strain field surrounding the

crack tip (20, 21). Thus, at least in theory, direct comparisons of stress corrosion crack growth in specimens of different geometries can be made. Also, a designer could conceivably predict the behavior of a stress corrosion crack in a service component from laboratory data, knowing the stress intensity solution for the crack in the component (18).

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Precracked specimens are exposed in suitable environments to determine the threshold stress intensity factor, K<sub>ISCC</sub>, to initiate stress corrosion crack growth at a relatively deep flaw and to measure SCC propagation rates (Figure 10). The threshold stress intensity can be used to calculate conditions (applied stress and crack depth) below which crack growth by SCC would not be expected, or would be negligibly slow in a given environment. This information could be very useful in design, and along with the SCC propagation rates, also can be used for ranking materials. A variety of specimen configurations and loading methods can be used, as described in detail by Smith and Piper (22).

Tests can be made with either (a) constant applied load, during which K increases until the crack grows to a critical length and the specimen fractures, or (b) constant deformation, during which K decreases as the crack grows to a length where it arrests, theoretically at  $K_{\rm ISCC}$  (or  $K_{\rm th}$  if LEFM requirements are not satisfied). Tests also can be made with specially contoured

specimens for which the K remains constant during crack growth. An example of comparative data from a K-increasing and K-decreasing tests of 7075-T651 plate is shown in Figure 11. Most of the SCC velocity vs. stress intensity factor (V-K,) data available in the literature for aluminum alloys have been obtained using double cantilever beam (DCB) test spcimens which were precracked by tension loading to "pop-in" (Figure 10b). Although good reproducibility of DCB  $V-K_T$  data has not been established, it is generally accepted that such data enable alumiinum alloys to be effectively ranked with respect to resistance to SCC growth (4, 18, 23). Hyatt and Speidel (18), have suggested that  $\text{V-K}_{\text{T}}$  data could be used to predict safe lives of components containing small stress corrosion cracks, but if these data are to be used in this way it is essential that the validity and accuracy of this procedure is established (19, 21, 24, 25).

There are experimental difficulties in the determination of threshold stress intensity factors. These difficultis are associated with the irregularities of initiation and growth of SCC, and the wedging action of corrosion products (4, 26). Although the actual incubation of SCC is not considered in the development of the V-K relationship, the initiation stage nevertheless is involved because it is necessary to develop intergranular SCC at the tip of the transgranular mechanical flaw (crack). The initiation time may be so short as to be negligible for materials with low resistance to SCC, but it can be quite

(4)

long for SCC-resistant materials. The formation of wedges of solid corrosion products can change the crack tip stress state, with the result that the actual stress intensity at the crack tip is higher than that calculated from the remote loading alone. Unfortunately, the effective K at the crack tip cannot be determined in the usual test, and since the calculated K is erroneous under weging conditions, a true Kth cannot be readily determined by this method. Examples of prolonged crack extension and crack tip stress intensity factor\* variation with time are shown for materials of four different degrees of resistance to SCC in Figure 12. (26) In recognition of the practical difficulties in estimating threshold stress intensity factors with bolt loaded DCB tests, more rigorous techniques have been proposed via the use of valid plane strain specimens stressed by constant load (26, 27).

## 2. Application of Elastic Plastic Fracture Mechanics (EPFM)

The LEFM approach to SCC testing has provided valuable insight into environment-assisted crack growth (both SCC and corrosion fatigue). However, the application of LEFM is limited to cases where a crack of substantial dimensions exists in a primarily elastic stress field. That is, the volume of high sress ahead of

<sup>\*</sup> Calculated from remote loading alone.

a crack in a loaded body (the region of confined plasticity) must be small relative to the length of the crack and geometric dimensions. Small cracks in small specimens do not adhere to these LEFM requirements, and the development of elastic-plastic fracture mechanics (EPFM) in the last decade has extended the range of the fracture mechanics approach to cases of more extensive plasticity. This should better enable the characterization of the mechanical driving force for small cracks in small specimens. The development of EPFM was motivated by the limitations of LEFM, especially with regard to very tough and ductile materials where very large specimens were needed to adhere to LEFM requirements. Also, it is qustionable whether LEFM requirements are satisfied when testing tough SCC-resistant materials under high applied loads. The EPFM analogy to K in LEFM is the J-integral. The crack driving force J was derived from a contour integral around a crack and was found to be independent of path even when a significantly large plastic zone (compared to other characteristic dimensions) exists ahead of the crack. This enables the determination of J, which is a crack-tip field characterizing parameter, to be made from measurements relatively far from the crack-tip. It has been shown that when conditions of LEFM are met, (i.e. limited plasticity) J and K are directly relatable (28). The application of EPFM thus extends the fracture mechanics approach to cases of large scale yielding ahead of a short crack. Although EPFM does have limitations which have not been fully explored, investigators have shown that the approach yields quite good predictions of crack driving force even in cases which are theoretically out of the validity range (28). Thus, the application of EPFM appears to be quite general and flexible.

An enticing approach to optimizing SCC testing techniques is to apply EPFM methodology to breaking load tests of smooth tension specimens. The use of breaking load tests is a promising new technique for evaluating SCC damage in statically loaded smooth specimens by performing tensile tests after various periods of exposure. This technique has been explored previously but has not been used to any appreciable extent except to calculate stress corrosion indexes (29, 30). Recent test results of B. M. Ponchel at Alcoa Laboratories have suggested practical advantages for this approach. Figure 13a shows schematically the effect of applied stress and time on the mean breaking strength after extended exposure. Conventional percentage failure of replicate specimens exposed for a critical exposure tilme, t, can be calculated from the mean breaking strength of replicate tests. Also, EPFM can be applied to calculate an effective SCC crack depth from the breaking load (stress), the material's mechanical properties, and a geometry-specific solution for the crack driving force.

Specimens can be removed and tension tested after various intervals of exposure to assess the rate of growth of SCC flaws, Figure 13b. Thus, the analysis of breaking load data with fracture mechanics may provide a quantitative means for tracking SCC damage with time. Exploitation of fracture mechanics concepts for use with the breaking load method will be attempted in Phase II of this contract.

#### D. SUMMARY

Smooth specimen SCC tests provide relative rankings of material performance under a given set of environmental conditions. Specimen time-to-failure includes both initiation and propagation stages of SCC, and traditionally the mechanical parameter used to interpret these data is the applied gross stress. Test results are dependent on mechanical aspects of the test, such as method of loading and specimen size, which can have variable effects on the initiation and propagation lifetimes and can infuence estimates of a threshold stress.

Mechanical acceleration of SCC can be achieved by dynamic loading under a constant strain rate. Choice of a critical strain rate, however, is dependent on the conditions of testing.

The conventional use of precracked specimens to study SCC propagation involves linear elastic fracture mechanics, which can provide a quantifiable mechanical crack driving force,  $\mathbf{K}_{\mathbf{I}}$ , for characterizing crack propagation.

Many technical limitations must be placed on the interpretation of SCC test data. An LEFM analysis is limited to relatively large cracks, for example, and can't generally be used to characterize shallow growth in small smooth test specimens. It is proposed that elastic-plastic analysis be used to extend the

fracture mechanics approach to smaller specimens and shallow cracks. This will be attempted in Phase II of the current contract by coupling fracture mechanics with a promising new accelerated test procedure, the breaking load method, which uses data from tension tests performed on replicate groups of exposed smooth specimens to rank SCC. It is believed that this approach can lead to more quantitative SCC characterization, better understanding of relationships between testing methods and optimization of test procedures.

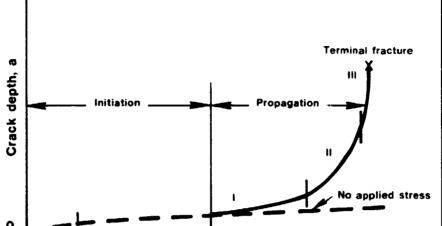
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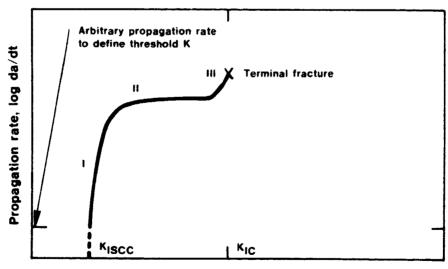
C



- A Localized breakdown of oxide film.
- **B** Formation of corrosion fissures, localized concentrations of stress, and nucleation of SCC.
- C Propagation of SCC in two or three stages with changing dependency on stress intensity factor.

Time

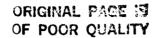
(a) Initiation and propagation of SCC.

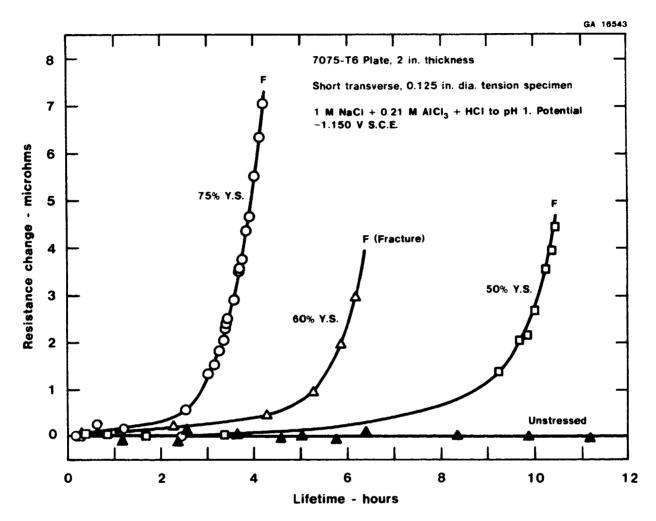


Crack tip stress intensity, K<sub>1</sub>

(b) Application of linear elastic fracture mechanics to propagation of SCC (Ref. 2).

Schematic Diagrams of the Initiation and Propagation of SCC. Figure 1



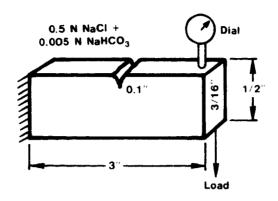


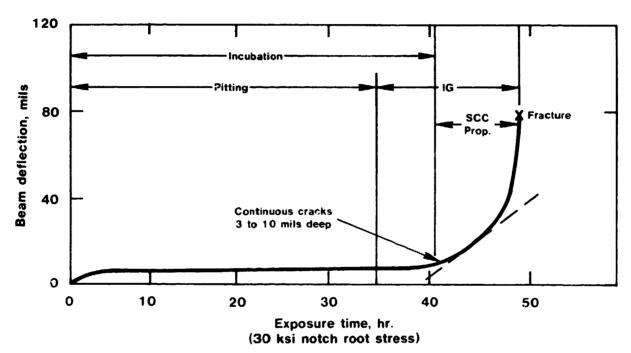
Smooth Specimen Life Curves Measured by Changes in Electrical Resistance. Note the Absence of Distinct Separation of Initiation and Propagation Stages (Ref. 1).

Figure 2

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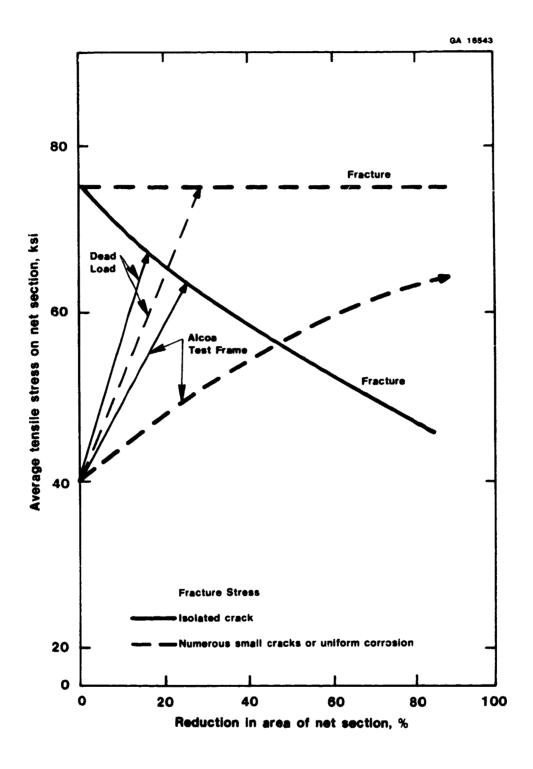
#### Short transverse tests 3 in. thick plate

5.7% Zn - 2.7% Mg - 1.35% Cu - 0.5 Mn - 0.3% Fe + Si (HT 3 hr. 465°C, BWQ, aged 24 hr. 120°C.)

95% of specimen life taken up by formation and growth of pitting and intergranular attack to a certain degree of acuity from which true SCC emanates (Ref. 3).

Notched Specimen Life Curve Shown by Beam Deflection.
Length of Time Required for the Formation of Intergranular SCC was Determined by Metallographic Examination of Individual Specimens Removed from Test after Various Periods of Exposure.

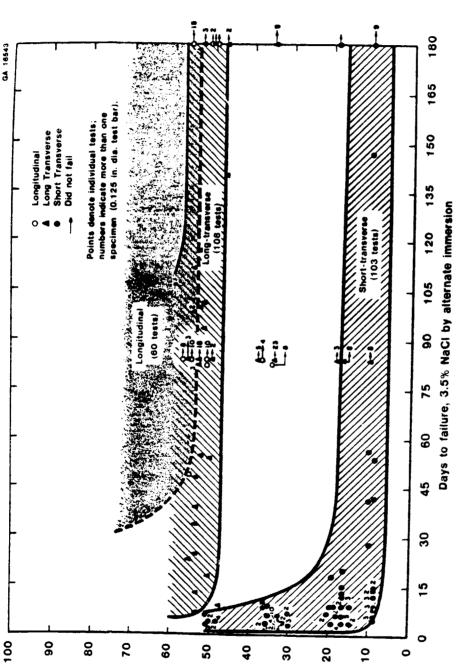
Figure 3



Effect of Corrosion Pattern on Fracture Stress and on Net Section Stress in 0.125 in. Dia. Aluminum Alloy Specimen (Ref. 4)
Figure 4

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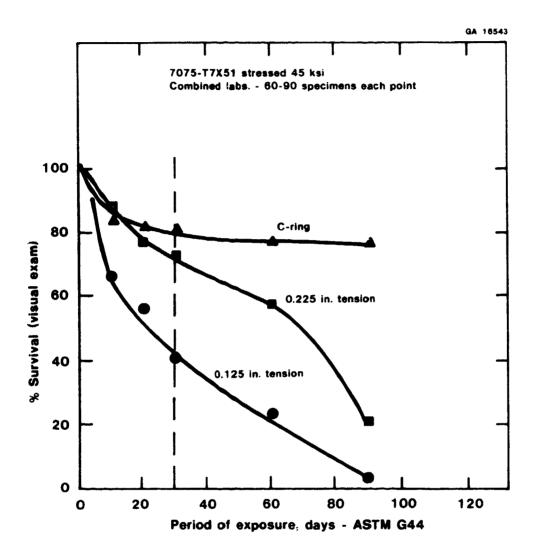


Sustained tension stress,

The data plotted in this graph represent a summary of a large number of tests of 7075-T6 plate, 1/4 to 3 in. thick. Bands were drawn to show the distribution of the stress corrosion failures of the long transverse and the short transverse specimens. Because of the very few failures of longitudinal specimens only the the bottom limit of a band was estimated.

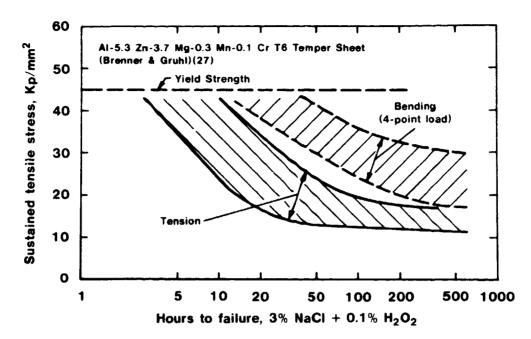
Resistance to Stress Corrosion Cracking of 7075-T6 Plate as Influenced by Direction of Stressing (Ref. 6). Figure 5

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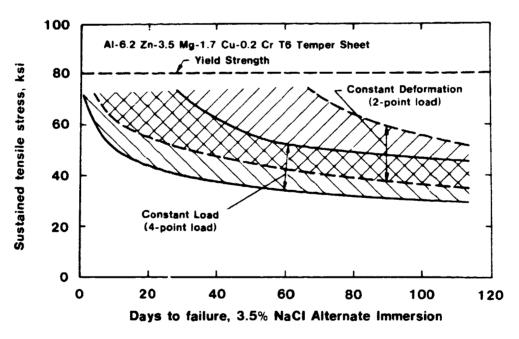


Influence of Specimen Configuration on Stress Corrosion Test Performance (Ref. 7).

Figure 6



(a) Comparison of direct tension versus bending, with constant load.

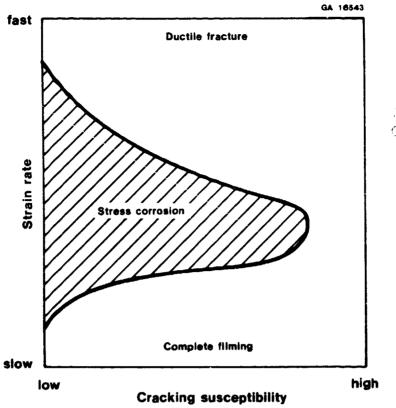


(b) Comparison of constant-load versus constant-deformation stressed beams.

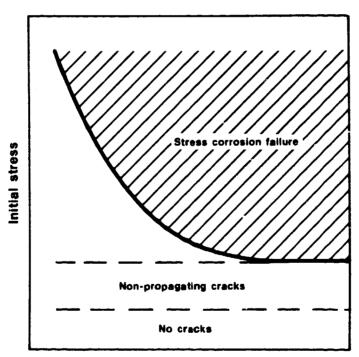
Influence of Methods of Loading on SCC Test Performance (9).

Figure 7



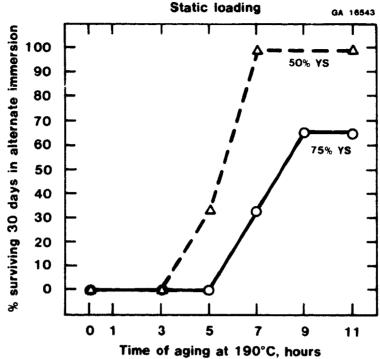




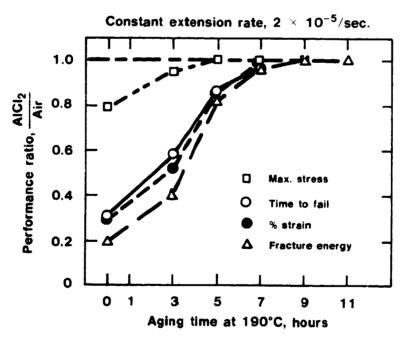


Failure time

Schematic Illustration of the Relationship Between Strain Rate, Applied Stress, and Sensitivity to Stress Corrosion Failure (Ref. 13). Figure 8



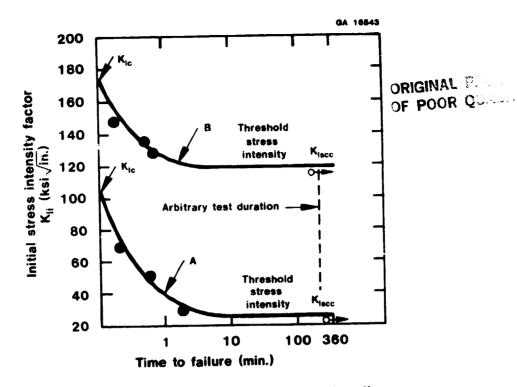
(a) Percent of specimens surviving 30 days in alternate immersion, at two applied stress levels, with increasing aging time at 190°C.



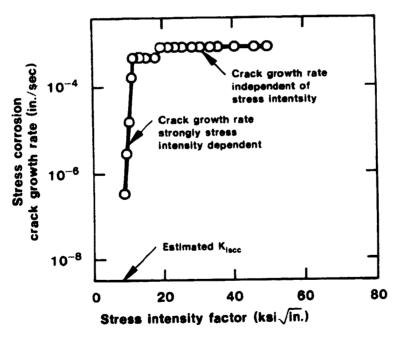
(b) Stress corrosion cracking performance ratios as a function of artificial aging time.

Determination of SCC Resistance of Incrementally Aged 2124-T351 Plate by Slow Strain Rate and Static Load Testing (Ref. 14).

Figure 9



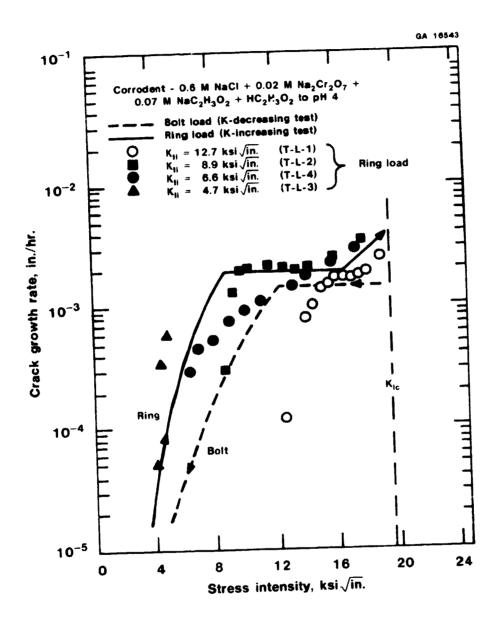
 (a) Typical stress corrosion characterization for alloy with low resistance (curve A) and high resistance (curve B) to stress-corrosion cracking.



(b) Typical relationship between applied stress intensity and crack growth rate for a commercial aluminum alloy.

Typical Fracture Mechanics (LEFM) Type SCC Test Results (Ref. 9). Figure 10

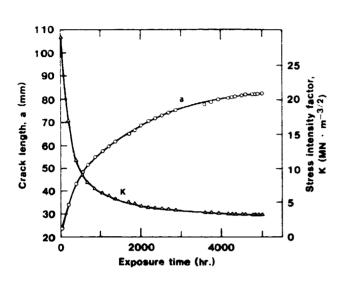
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K-Rate Curves from Ring Loaded (K-increasing) and Bolt Loaded (K-decreasing) Fatigue Precracked Compact Specimens of Plate Alloy 7075-T651 Loaded in the S-L Direction (Ref. 4).

Figure 11

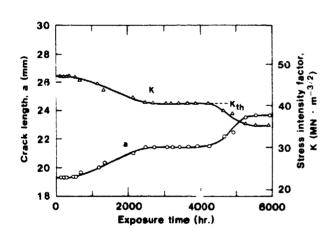
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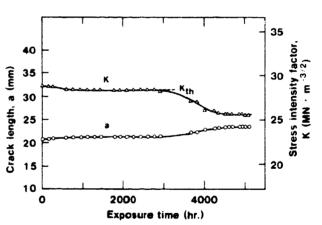


25 (MN) = 32 (Stress intensity factor, K (MN) = 3.2)

(a) 7075-T652 plate, 30 mm thick.

(b) Zergal 4-T-(H)  $A_1A_3$  forging section, 60  $\times$  30 mm.





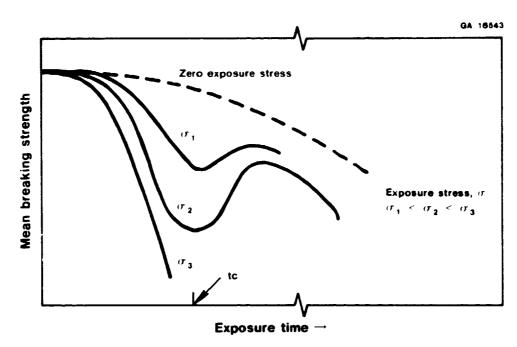
(c) 7175-T7652 forging section (produced with ITMT), 30  $\times$  110 mm.

(d) 7075-T7352 plate, 30 mm thick.

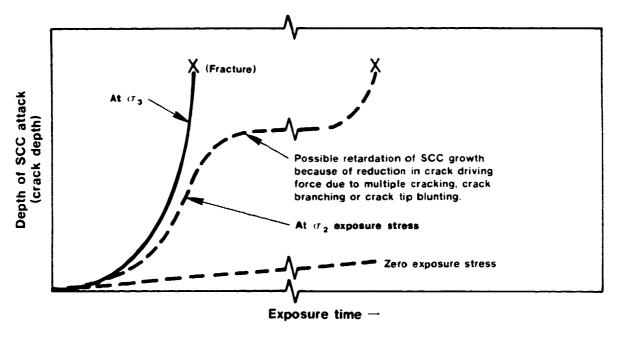
Examples of Various Crack Growth Behavior and Associated Stress Intensity Factors Obtained with DCB Specimens for Different Materials Using Arbitrary Cut-off Exposures to Obtain Estimates of K<sub>th</sub>, Independent of the Influence of Corrosion Product Wedging (Ref. 26).

Figure 12

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(a) The effect of prior exposure on the breaking strengths of initially smooth tensile bars.



(b) The development of SCC with length of SCC exposure.

Schematic Representation Showing How Development of SCC Relates to the Breaking Strength of Pre-Exposed Tensile Bars.

Figure 13

IV. ENVIRONMENTAL ASPECT OF STRESS CORROSION TESTING FOR ALLOY DEVELOPMENT AND SELECTION

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D. O. Sprowls

Fourth Technical Progress Report Submitted in Partial Fulfillment of:

NASA CONTRACT NAS1-16424 - A STUDY OF ENVIRONMENTAL CHARACTERIZATION OF CONVENTIONAL AND ADVANCED ALUMINUM ALLOYS FOR SELECTION AND DESIGN

PHASE I REVIEW OF THE LITERATURE

Reported for: Contract Period Ending April 30, 1982

# A. INTRODUCTION

For stress-corrosion cracking (SCC) to occur, there must be interaction of appropriate combinations of chemical and electrochemical conditions in the environment with a specific metallurgical condition of the metal and a requisite level of tension stress (1). Laboratory experiments have shown that mere traces of water may provide a sufficient environmental condition to promote SCC in highly stressed specimens of susceptible Al-Ma (5XXX) and Al-Zn-Mg (7XXX) alloys (2, 3). An example of the accelerating effect of increasing water content in air on the SCC growth in highly stressed short transverse specimens of 7075-T651 is illustrated in Figure 1 from the work of Speidel (3). Growth of SCC in humid air is further accelerated by the usual contaminants present in the atmosphere at seacoast and inland industrial sites, as can be seen by a comparison of the plateau velocities in Figure 2 (4). It is in atmospheric environments such as these that most cases of SCC occur in the service of commercial high strength aluminum alloys.

It is generally recognized that environmental variables can have profound effects, either detrimental or beneficial, on tendencies of stressed components to crack. Each one or a combination of these factors can affect both the thermodynamic and the kinetics of the electrochemical processes that control SCC. Thus, choice of environmental conditions provide an important basis for developing accelerated SCC test procedures.

### B. SPECIFICITY OF ENVIRONMENT-ALLOY COMBINATIONS

The specificity of environment-alloy combinations makes general predictions of the expected SCC behavior of alloys somewhat tentative. Although less uncertainty is involved when the metallurgical structures and their electrochemical characteristics are established, the present state-of-the-art does not provide scientific models for calculating the estimated risks of material serviceability. Therefore, appropriate stress corrosion tests are needed; but tests can be misleading if they are not properly related to the alloy, temper and environment of interest.

The following laboratory experiment illustrates the effect of alloy chemistry on the SCC behavior in several different aqueous solutions (1). Highly stressed short transverse specimens representing three different types of susceptible alloys (based on typical behavior in a seacoast atmosphere) were exposed in triplicate to six neutral solutions of one-normal sodium salts. It can be seen from the bar graphs in Figure 3 that the Al-Cu alloy (2219-T37) failed only in the sodium chloride solution, and the Al-Zn-Mg-Cu alloy (7075-T651) failed in both sodium chloride and sodium bromide, but at least one of the three specimens of the Al-Zn-Mg alloy (7039-T63) stress corrosion cracked in all of the solutions. It is noteworthy that there were no failures of the SCC-resistant 2219-T87 and 7075-T73 specimens. The tendency for Al-Zn-Mg alloys containing relatively low copper (7079, 0.7% Cu) or no copper (7039) to be susceptible to SCC in a wide variety of

mildly corrosive atmospheres is well known from service experience as well as from laboratory tests (1, 5, 6). Al-Zn-Mg-Cu alloys with higher copper contents (7075, 1.6% Cu; 7178, 2.0% Cu, 7050, 2.2% Cu) are less vulnerable, and Al-Cu alloys (2XXX) are still less susceptible. Example of these tendencies are shown in Tables I and II for smooth tension specimens exposed at various stress levels to atmospheric environments (7).

The importance of alloy-environment specificity on SCC evaluation will be touched on again in the following sections.

## C. FIELD TESTING AND SERVICE ENVIRONMENTS

A field test is one in which a metal specimen is placed in an environment where conditions simulate those anticipated in the service of a structure. Typical examples are immersion in seawater, exposure to the atmosphere at marine or industrial sites, chemical plant streams, etc. Field tests might be performed with test coupons or with actual or simulated structural components.

The following example illustrates the value, and in some cases the necessity of exposure tests performed in the actual service environment as an adjunct to laboratory evaluation. In this example, the standard 3.5% NaCl alternate immersion test data for 2024 and 7075 alloy proved to be of no use in predicting serviceability of these alloys for handling rocket propellant

(4)

oxidizers such as nitrogen tetroxide and inhibited red fuming nitric acid (IRFNA) (1). The alternate immersion test had shown 2024-T351 and 7075-T651 to be susceptible to SCC at low short transverse stresses, whereas 2024-T851 and 7075-T351 were quite resistant, and these performances were borne out by outdoor field tests in seacoast and industrial atmospheres. However, in proof tests with exposures to IRFNA at 74°C (165°F), the actual service environment, SCC occurred in both tempers of 7075 alloy, and did not occur in either temper of 2024 alloy (Figure 4). It was gratifying, however, that there were no inexpected failures with the 2219-T87 and 6061-T651 materials.

### D. ACCELERATED TEST MEDIA

For most purposes, it is expected that a short exposure in an accelerated test will reliably and accurately predict the SCC performance of an alloy over a long period of service. In order to meet this prime function of the accelerated test, it is necessary that the test conditions be selected with due regard to the service to which the metal will be subjected. An important requirement of the accelerated test is that it be capable of duplicating the in-service failure mechanisms when such experience is available (8). This problem is complicated because it involves not only the consideration of an appropriate environment, but also the knowledge of realistic types of mechanical loading and stress magnitudes. This task can be complex in situations for which there is no past experience.

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Hyatt and Speidel in a major stress corrosion program of the 1960's (9) investigated SCC propagation rates for 7075-T651 and 7079-T651 materials under short transverse stress in a wide variety of chemical environments encountered in aircraft service. One very significant finding was that the chloride, bromide, and iodide ions are unique in their ability to accelerate SCC growth in neutral solutions above and beyond the velocity measured in distilled water. This is illustrated in Figure 5. None of the other anions listed showed any tendency to accelerate SCC even under extreme metallurgical, mechanical and electrochemical conditions. It is noteworthy that chloride, bromide and iodide ions also are the unique pitting agents for aluminum alloys and accelerate crevice and intergranular corrosion. Therefore, it would be expected that they can influence not only propagation, but also initiation of stress corrosion cracks. Chloride solutions historically have been favored for accelerated tests because sodium chloride is widely distributed in nature, and the test results are relatable to SCC behavior in natural environments, particularly where there are strong marine influences.

Hyatt and Speidel (9) also observed sign licant SCC growth rates for 7075-T651 in a variety of off-the-shelf organic solvents, aircraft flight fuel, engine oil and hydraulic fluids. These data are shown in Figure 6. It was noted that the plateau velocities measured in the organic solvents fell within the scatterband for SCC tests in water. This observation is consistent with the hypothesis that it is the small water content

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of the commercial solvents which causes SCC. The SCC crack growth in the flight fuel, engine oil and hydraulic fluids was lower, although still significant, and was about the same as in moist air with about 30% relative humidity (refer to Figure 1). It also was found that halide additions to organic solutions can greatly accelerate SCC in 7075-T651 and 7079-T651 alloys.

Acceleration of SCC growth by chloride, bromide and iodide depends in a complex way on metallurgical, mechanical, electrochemical and other environmental parameters which must be controlled if a meaningful quantitative SCC test is to be attempted. For example, Speidel (3, 6) observed that the SCC velocity for 7075-T651 in water could be increased only by a factor of four by sodium chloride additions. However, with 7079-T651 alloy, the same change in environment caused a 1000-fold increase in the SCC plateau velocity, thus showing that the SCC acceleration by halides also is influenced by metallurgical (composition) parameters.

The extensive investigations by Hyatt and Speidel (9) and subsequent studies by Brown, Foley and associates (10-12), in which SCC in very susceptible alloys such as 7075-T651 and 7079-T651 was measured in terms of crack growth rate, have clearly demonstrated the importance of a number of parameters that must be controlled in accelerated SCC evaluation tests. The following procedures have been shown to be effective ways to accelerate SCC growth in aqueous halide solutions, with variable results

depending upon the nature of the test material and the mechanical techniques:

- (a) Increase the anion concentration.
- (b) Increase acidity (lower pH).
- (c) Increase temperature Especially effective for Al-Zn-Mg alloys.
- (d) Add oxidizer: Simple aeration of the solution, or addition of oxidants such as hydrogen peroxide nitrates, chromates - Especially effective for Al-Cu, Al-Mg and Al-Zn-Mg-Cu alloys.
- (e) Careful control of applied potential.

Accelerated SCC testing can be very complex, as stated previously, and for additional clarification it is suggested that readers study the references listed at the end of this section. Some of these works contain significant implications regarding stress corrosion mechanisms. The difficulty of identifying a single accelerated test medium for all aluminum alloys, or of even finding the optimum corrodent for a given alloy, can be illustrated by the following examples taken from Alcoa testing experience.

Although nitrates and sulfates dissolved in water tend to retard rather than to accelerate SCC, their presence in chloride environments can produce a synergistic stimulation of intergranular corrosion and SCC (13, 14). This effect has been observed at sites such as in the city of Los Angeles where the atmosphere contains a disproportionately high content of  $NO_2$  compared to that at Point Judith, RI, and New Kensington, PA (14).

The percent survival data in Figure 7 showing the relatively poor performance in Los Angeles were obtained with small sized axially loaded tension specimens which are highly influenced by the initiation of localized corrosion and SCC, as well as by growth rate of the SCC. When similar materials were tested with mechanically precracked double cantilever beam (DCB) specimens in which only the growth of SCC was monitored (Figure 8), the performance of the 7075-T7351 material at Los Angeles was not adversely affected by the NO<sub>2</sub> contaminant—in fact, the performance at Los Angeles was better than at Point Judith and similar to that in New Kensington. Thus, assessment of the effects of environmental chemistry can be markedly influenced by other factors, such as climatic conditions, the type of test specimen and the method of measuring damage due to SCC.

The smooth specimen data in Tables I and II indicate the difficulty with trying to use a single test such as the 3.5% NaCl alternate immersion test to characterize the SCC behavior of all types of alloys. Test results in Table II for the Al-Zn-Mg alloy X7106-T63 indicate that the boiling 6% NaCl test would be more realistic for this type of alloy although it does not look promising for Al-Cu type of alloy (2025). These observations are in accord with other unpublished Alcoa testing experience.

The ultimate determination of the validity of an accelerated SCC test medium requires a correlation with the results of service experience or with the results of appropriate field tests (8).

Unfortunately, to be meaningful in the instance of some alloys, exposure in service environments can require many years. A specific example of the correlation of two accelerated test media with a service environment or a copper-free Al-Zn-Mg alloy (7039) is shown in Figure 9. These data demonstrate that the 4-day boiling 6% NaCl test relates better to the industrial atmosphere exposure than either a 90-day or an 180-day exposure to the 3.5% NaCl alternate immersion test (which with an 180-day test period, could hardly be considered accelerated). The data also illustrate that in a service environment the length of exposure required to demonstrate the SCC behavior of an alloy can require a number of years, a circumstance which complicates correlation tests.

while it is recognized that the local environment generated inside a crevice or stress corrosion fissure can be quite different from the bulk environment, detailed knowledge of "crack-tip" chemistry and reaction kinetics still is speculative. Knowledge of this type is required before quantitative predictive models of SCC performance can be developed.

### E. RECOMMENDED TEST MEDIA FOR SPECIFIC ACCELERATED TESTS

United States is in its infancy, with the first standards published by ASTM being for test specimens, which can be used with any metal and most environments. These standards are for smooth specimen tests (G30, G38, G39, G49) (15). The first environmental standard practice for aluminum alloys were published in 1975 and

that was ASTM G44, "Standard Practice for Alternate Immersion Stress Corrosion Testing in 3.5% Sodium Chloride Solution" (15). Then G47 was published in 1976 with specific conditions of specimen types and exposure periods for two types of aluminum alloys: Al-Cu (2XXX) with 1.8-7.0% copper, and Al-Zn-Mg-Cu (7XXX) with 0.4-2.8% copper. Following this in 1980 was G64, "Standard Classification of the Resistance to Stress-Corrosion Cracking of High-Strength Aluminum Alloys," based on service experience and smooth specimen tests made according to ASTM G47. These are the only widely accepted environmental standards for aluminum alloys at present. There are some tests, not in use in this country, prescribed in certain European specifications.

### 1. Smooth Specimen Tests

### a. 3.5% NaCl Alternate Immersion Test (ASTM G44)

This test is specified in G47 for testing high strength 2XXX and 7XXX (0.4-2.8% Cu) alloy with standard smooth specimens, but is commonly used as an all purpose test for other types of aluminum alloys. It is the accelerated test method most widely used in the U.S.A. for evaluating the SCC resistance and is called out in various materials specifications. A disadvantage of the 3.5% NaCl corrodent is the severe pitting that develops in certain high strength alloys. This is particularly a problem with copper-bearing alloys when tested with specimens of small crosssection. An allowable alternative in G44 for the 3.5%

NaCl solution is Substitute Ocean Water (without heavy metals) prepared per ASTM Specification D1141. The advantage of this corrodent is that it causes less pitting corrosion than the plain sodium chloride solution. The ASTM task group (G01.06.91) for Stress Corrosion Testing Aluminum Alloys is collecting comparative test data for the two test media. There are some indications that the Substitute Ocean Water may not be as aggressive in causing SCC.

Mr. T. S. Humphries of NASA Marshall Space Flight Center has proposed a more practical alternative for the Substitute Ocean Water which appeared promising on the basis of limited tests (16). This new test medium contains 2.86% sodium chloride and 0.52% magnesium chloride, the same chloride content as in sea water. Additional evaluation of this test medium is needed.

Another way to circumvent the pitting problem with the 3.5% NaCl solution is by the use of optimized (shorter) exposure periods, such as determined by the breaking load test method described in the Phase II report of this contract.

### b. Boiling 6% Sodium Chloride (Continuous Immersion)

This rapid (4-day) test is the one most generally used by U.S.A. aluminum producers to evaluate the SCC behavior of copper-free 7XXX type aluminum alloys via conventional smooth specimen test procedures. A sample of test results favoring this approach are shown in Table II and Figure 9. An ASTM standard is currently being drafted for this test medium.

### 2. Tests with Fracture Mechanics Type Specimens

At present, there are no standards for test media to be used with precracked specimens. A periodic moistening procedure (dropwise application of 3.5% NaCl solution three times a day) devised by Hyatt (17) as a substitute for the alternate immersion procedure used for smooth specimen testing has had some usage by other investigators. This technique produces considerably more rapid growth of SCC in both Al-Cu (2024-T351) and Al-Zn-Mg-Cu (7075-T651) susceptible alloys than continuous immersion in 3.5% NaCl (Figure 10) (18). A previous NASA contract program carried out at Alcoa Laboratories (18) showed that the Hyatt (Boeing) procedure ranked SCC growth of various aluminum alloys in the same order as exposure in a seacoast atmosphere (Figures 11 and 12). The ranking in an inland industrial atmosphere was the same for the alloys except the sensitized Al-Mg (5456) which showed a marked reduction of crack growth in the latter environment.

corrosion product wedging effects were noted after extended exposure to the salt solution and the seacoast atmosphere. In subsequent investigations of high strength Al-Zn-Mg-Cu alloys (19, 20), exposure to substitute ocean water by alternate immersion produced alloy rankings similar to those in atmospheric exposure with decidedly less evidence of corrosion product wedging. Possibly the Humphries NaCl/MgCl<sub>2</sub> solution could also be advantageously used for these types of tests.

### 3. Slow Strain Rate Tests

There are no standards for this new testing approach. Various solutions have been used in additions to plain 3.5% sodium chloride. Because 3.5% salt solution may not be aggressive enough for the slow strain rate testing approach, more corrosive test media considered include oxidant additions to the sodium chloride solution or more acidic solutions such as aluminum chloride (21, 22). In a European round robin testing program conducted by the EAA Working Party (23) using a variety of aluminum alloy types and several corrodents, found a solution containing 3% NaCl + 0.3% H<sub>2</sub>O2 to be the most promising test medium considered for possible standardization. A second promising solution was 2% NaCl + 0.5% Na<sub>2</sub>CrO<sub>4</sub>, pH3.

### F. SUMMARY

- 1. Traces of water (vapor or liquid) constitute a sufficient environment to promote SCC of susceptible 5XXX and 7XXX series alloys. Contaminants in seacoast and inland industrial (urban) atmospheres may accelerate the SCC process in susceptible aluminum alloys.
- The halide ions (chloride, bromide and iodide) stimulate pitting of aluminum and SCC of susceptible alloys.
- 3. Chloride solutions historically have been favored for accelerated SCC tests because sodium chloride is widely distributed in nature, and the test results are relatable to SCC behavior in natural environments.
- 4. Choice of the appropriate environment for an accelerated SCC test is important, and difficult because of unique electrochemical interactions involving alloy microstructure and the many environmental factors that must be controlled.
- 5. It is necessary that accelerated SCC test conditions be selected with due regard to the intended service application. This consideration is important for alloy development programs as well as for the purpose of materials selection.

- 6. The present state-of-the-art does not provide scientific models for estimating risks of serviceability of alloys and tempers with regard to SCC. Important information that still is speculative involves the chemistry at the tip of a stress corrosion crack.
- 7. The ultimate validity of an accelerated SCC test rests on correlation with service experience or with the results of appropriate field tests.
- 8. Standardization of environmental conditions is needed for specific alloy systems subjected to the various types of SCC tests.

## 4

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COMPARISON OF RELATIVE SCC PERFORMANCES OF SEVERAL HIGH STRENGTH ALUMINUM ALLOYS IN 20-YEAR ATMOSPHERIC EXPOSURE AT DIFFERENT ENVIRONMENTS (1)

INLAND INDUSTRIAL AT NEW KEN., PA 3.5% NAC! ALTERNATE IMMERSION (4)	DAYS TO FAILURE		15,21 2 0K 84 2 0K 84	F0 40 7		2 0K 84	5,5	19, 3 0K 84	4 OK 84		5 0K 84
3.5% N	F/N (3)		2/2	7/0	2/2	2/2	2/2	1/4	0/4		5/0
IDUSTRIAL AT NEW KEN., PA	DAYS TO FAILURE		98,98,116 126,172,192,197 640,673,076	0.5.5.9.0	192, 192, 198	219,258,258,258 2376,2639,4689	205, 623, 1629	OK 7540	0K 7540		4 OK 7540
INLAND I	F/N (3)		3/3	5/3	3/3	4/4 3/3	3/3	0/4	0/3		0/4
SEACOAST AT PT. COMFORT, TX	DAYS TO FAILURE		59, 79, 88	1054,1054,2529	40,79,79	88,96,104,165 1090,4446,5997	40, 40, 45	96,96,137,508	2529,2 0K 7360		5 0K 7360
	F/N (3)		3/3	3/3	3/3	4/4 3/3	3/3	4/4	1/3		9/0
EXPOSURE SEACOAST AT PT. JUDITH, RI	F/N (3) DAYS TO FAILURE		27,27,56	653, 1284, 3605	27,27,49	49,76,90,208 6122,6331,0K 7510	49 56 56	56, 161, 252, 548	3 OK 7510		5 OK 7510
	F/N (3)		3/3	3/3	3/3	4/4 2/3	1/1	4/4	0/3		9/0
		Σ	33	<b>-</b>	55	37 18	45	2 00	22		44
	STRESS (2)	LE ALLO	75 50 50	52	75	22 22	75	20.	52	ALLOYS	75
	TEMPER (5)	SUSCEPTIBLE ALLOYS	7079-1651		7178-7651		2014-1651			RESISTANT ALLOYS	7075-17351 75

Transverse 0,125 in, diameter tension specimens machined from 2.5 in, dia, rolled rod (J.O. XA-179, NASA Contract NAS 8-5340) (Ref. 7). Stressed in Alcoa constant strain wedge type stressing frames. F/N denotes number of SCC failures/number of replicate specimens exposed. Similar to ASTN 644 except salt solution was made with commercial grade sodium chloride and New Kensington tap water. £88£9

4 OK 7540

Composition:

>	;	;	:	:	0.10
72	;	;	;	;	0.17
F	0.03	0.0	0.02	0.04	90.0
5	0.15	12.0	0.01	0.19	0.01
Zu	4.59	6.72	0.04	5.68	0.03
S.	3.48	2.60	0.57	2,35	0.0
£	0.20	0.02	0.82	0.02	0.29
55	0.10	0.10	0.88	0.10	0.1
a e	0.23	0.16	0.26	0.21	0.18
3	0.64	1.97	4.41	.54	6.28
Alloy	7079-1651	7178-1651	2014-7651	7075-17351	2219-187

I I

COMPARISON OF RELATIVE SCC PERFORMANCES OF SEVERAL HIGH STRENGTH ALUMINUM ALLOYS
IN 18-YEAR ATMOSPHERIC EXPOSURE AT DIFFERENT ENVIRONMENTS (1)

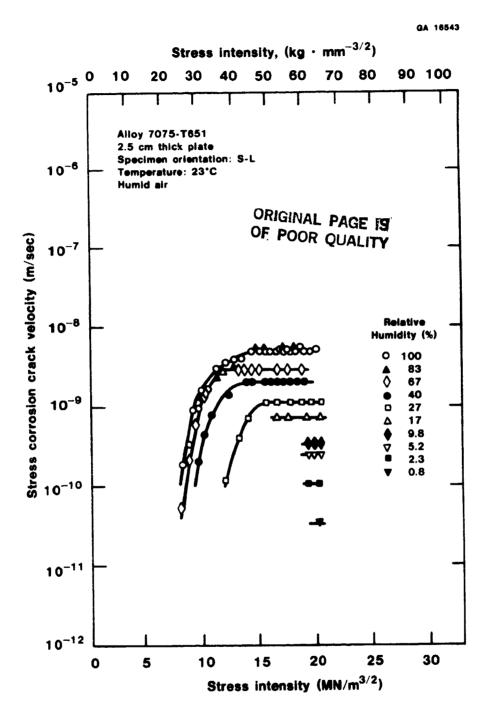
BOILING 6% NaCI	F/N (3) HOURS TO FAILURE	3/3 0.2,0.2,0.2 3/3 0.3,0.3,0.5 3/3 0.8, 1, 1	0/3 3 0K 96 0/3 3 0K 96 0/3 3 0K 96	Not Tested Not Tested
NALTERNATE IMMERSION (4)	DAYS TO FAILURE	5,11,78 11,78,0K 84 3 0K 84	2,3,4 2,4,4 5,2 0K 84	3 0K 84 3 0K 84
3.5% NaCl	F/N (3)	3/3 2/3 0/3	3/3 3/3 1/3	0/3 0/3
MLAND INDUSTRIAL AT NEW KEN., PA 3.5% NAC! ALTERNATE IMMERSION (4)	) DAYS TO FAILURE	15,15,15 17,27,31 31,45,115	3 0K 6635 3 0K 6635 3 0K 6635	3 0K 6635 3 0K 6635
INLAND	F/N (3)	3/3 3/3 3/3	0/3 0/3 0/3	0/3 0/3
SEACOAST AT PT. JUDITH, RI		90,115,115 115,115,115 115,115,485	115,375,0K 6600 3 0K 6600 3 0K 6600	3 OK 6600 3 OK 6600
SEACOAST	F/N (3)	3/3 3/3 3/3	2/3 0/3 0/3	0/3 0/3
SURE	STRESS (2)	40 27 13	27 20 13	40
ExP0	STRES.	25 25 25	±8.3¥	75 50
	ALLOY AND TEMPER (5)	X7106-T63	2025-16	7075-173

Short transverse 0.125 in, diameter tension specimens machined from die forged impellers (J.O. RE-741, Alcoa unpublished data). Stressed in Alcoa constant strain wedge type stressing frames. F/N denotes number of SCC failures/number of replicate specimens exposed. Similar to ASTM 644 except salt solution was made with commercial grade sodium chloride and New Kensington tap water. NOTES:

Composition:

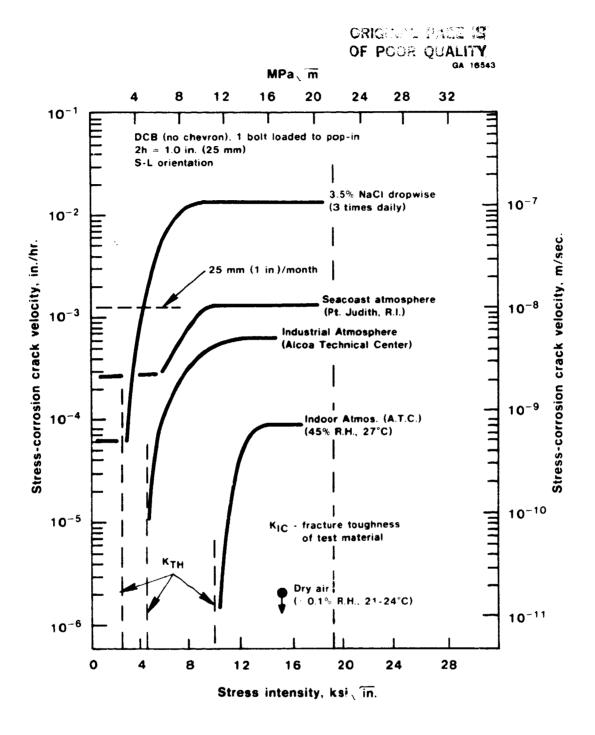
0.12 0.02 0.09 0.00 0.19 4.27 0.00 5.75 2.33 0.00 2.50 0.29 돌 0.09 0.76 0.10 0.12 0.23 0.20 0.0 Alloy X7106-T63 2025-T6 7075-T73

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Effect of Humidity and Stress Intensity Factor on Stress Corrosion Crack Velocity of High-Strength Aluminum Alloy 7075-T651 in Air (after Speidel, Ref. 3).

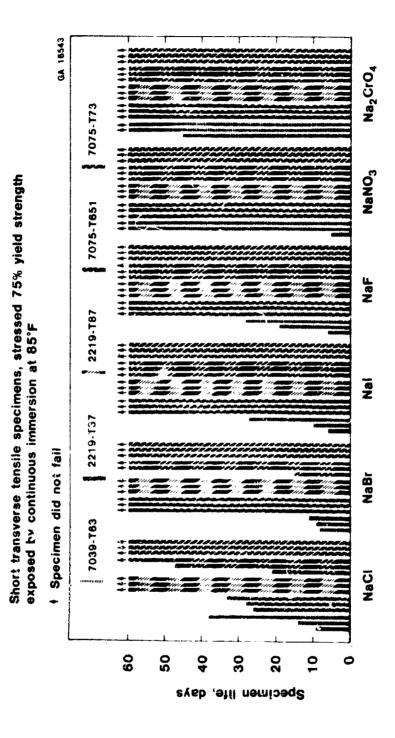
Figure 1



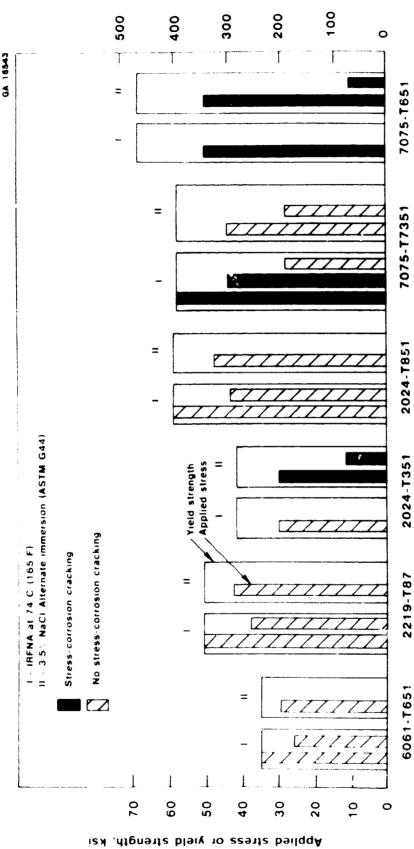
No SCC occurred during three years' exposure to dry air in a desiccator; however, the "plateau velocity" (horizontal part of each curve) and the apparent threshold stress intensity ( $K_{TH}$ ) varied with the environment.

Effect of Corrosive Environment on SCC Propagation Rate in 7079-T651 Plate, 64 mm (2.5 in.) Thick, Stressed in the Short Transverse Direction. Figure 2

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Stress Corrosion Cracking of Aluminum Alloys in Neutral Aqueous Solutions (Ref. 1). Figure 3



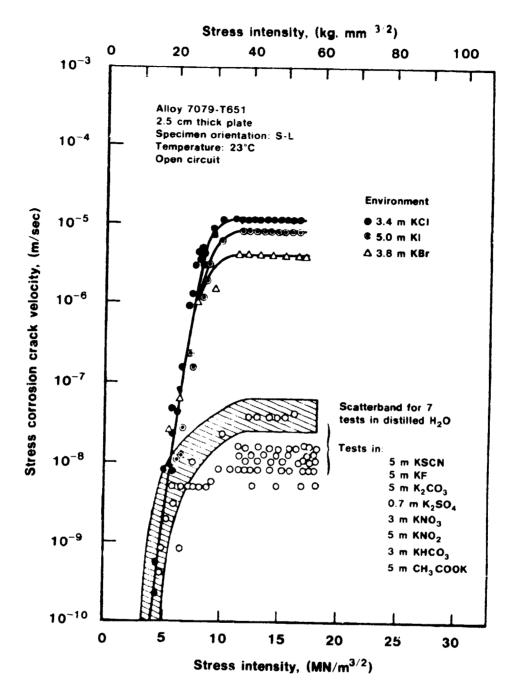
C-2

89

Pa

Each bar graph represents an individual short transverse c-ring test specimen (ASTM G38) machined from rolled plate and stressed at the indicated level.

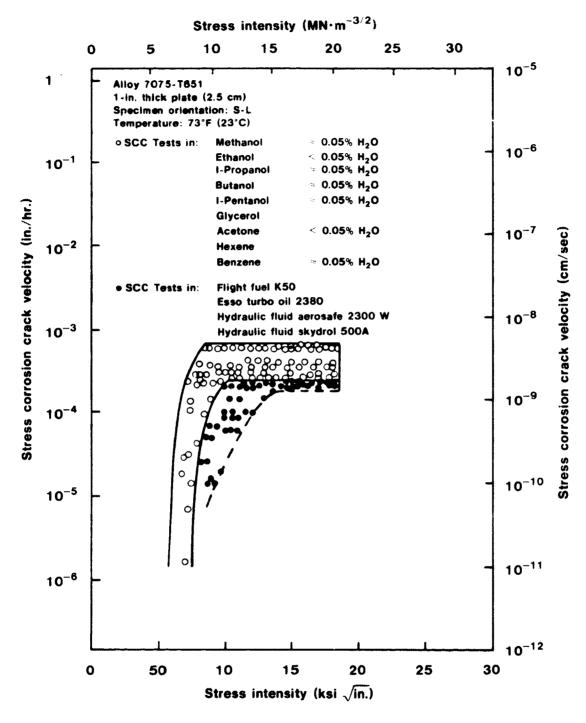
# SCC Resistance of Various Aluminum Alloys in Inhibited Red Fuming Nitric Acid (IRFNA) vs. Alternate Immersion in 3.5% Sodium Chloride Solution Figure 4



Influence of Various Anions on Stress Corrosion Crack Velocity of a High Strength Aluminum Alloy 7079-T651 Immersed in Various Aqueous Solutions (after Speidel, Ref. 3).

Figure 5

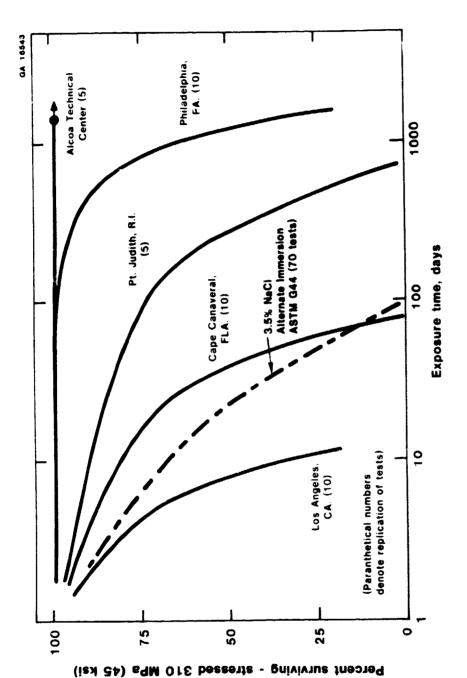
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Effect of Flight Fuel K50, Turbo Oil, Hydraulic Fluids, and Other Organic Liquids on Stress Corrosion Crack Growth of the Al-Zn-Mg-Cu Alloy 7075-T651 (after Speidel and Hyatt, Ref. 9).

Figure 6

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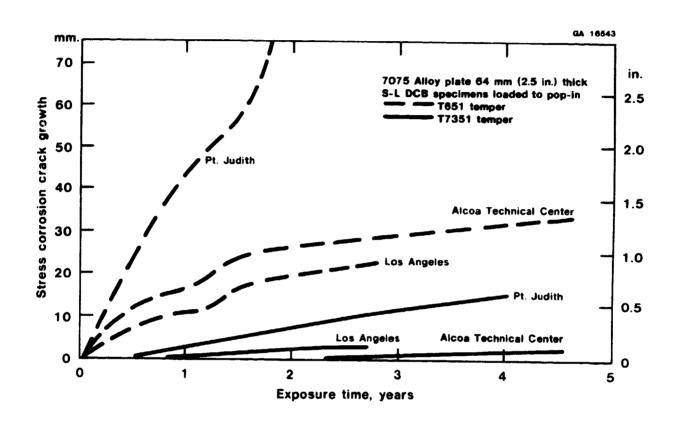


Tests were made on short transverse 3.18 mm (0.125 in.) diameter tension specimens from the same lot of 7075-T7651 type plate (Ref. 4).

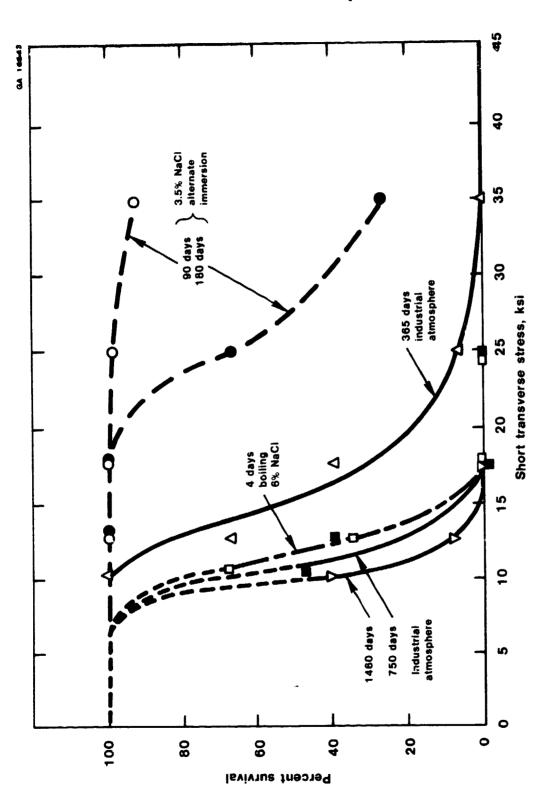
Effect of Variations in Geographic Atmospheric Environment on the Probability and Time to Failure by SCC of a Material with an Intermediate Susceptibility Figure 7

**(1)** 

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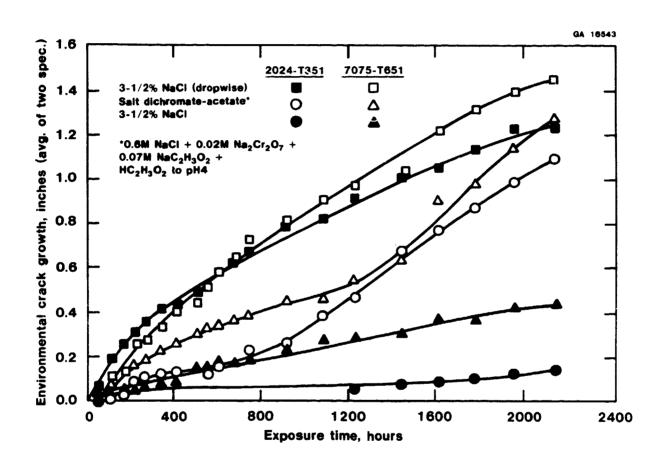


Comparison of SCC Growth in 7075 Alloy Plate for Various Geographical Locations within the Continental United States Figure 8



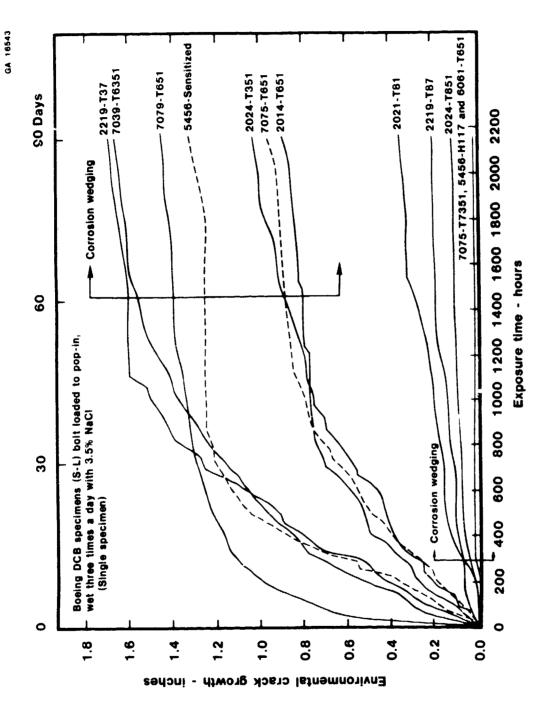
Correlation of Accelerated Test Media with Service Environments. Combined Data Shown for Five Lots of Aluminum Alloy 7039-T61 and T63 (4.0 Zn-2.8 Mg-0.3 Mn-0.2 Cr) Rolled Plate.

ORIGINAL PROTECTION OF POOR QUALITY



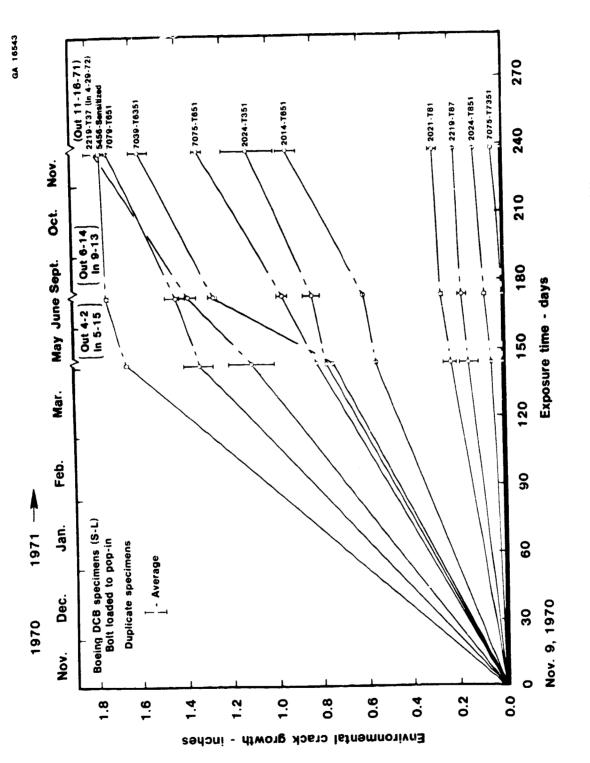
Environmental Crack Growth in S-L DCB Specimens (Boeing Design)
Bolt Loaded to Pop-in and Exposed to Various Corrodents.
Figure 10

ORIGINAL PACE IS OF POOR QUALITY



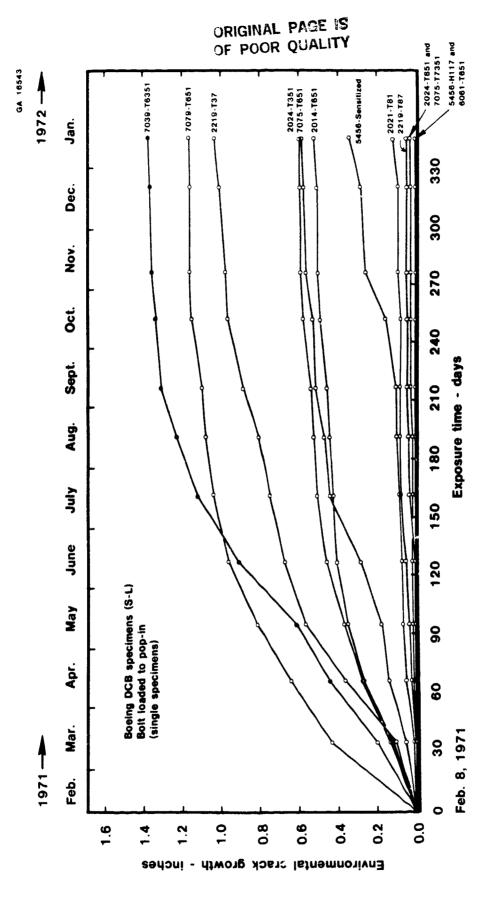
Environmental Crack Growth of Various Aluminum Alloys in 3.5% NaCl (Ref. 18). Figure 11

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Environmental Crack Growth of Various Aluminum Alloys in a Seacoast Atmosphere (Ref. 18). Figure 12





Environmental Crack Growth of Various Aluminum Alloys in an Industrial Atmosphere (Ref. 18) Figure 13

### V. CONCLUDING REMARKS

Various mechanical, environmental and applied aspects of SCC testing are reviewed separately in preceeding sections of this report, each having its own set of conclusions. A few major impressions of findings from this review and on state-of-the art SCC testing of aluminum alloys are given below.

- There are experimental difficulties associated with each of the various accelerated SCC testing techniques presently in use. These difficulties compound the task of characterizing degrees of susceptibility among alloys with relatively high resistance to SCC.
- There is still a need for improved accelerated test procedures, and preferably ones able to provide quantitative data which can be used to assess life of actual parts.
- 3. It is necessary that accelerated test conditions be selected with due regard to the service to which the metal will be subjected. This is an important consideration in testing for alloy development and material selection.
- 4. More definitive analyses of in-service applications are needed so that realistic SCC behavior targets can be set for alloy development and test methods can be selected to provide the most directly applicable data.

- 5. The present state-of-the-art testing does not provide scientific models for estimating risks of serviceability of alloys and tempers with regard to SCC.
- 6. A promising new accelerated test technique (the breaking load method) involving statically loaded smooth tension specimens permits meaningful statistical treatment of the test results, and offers the possibility of new practical interpretations of SCC in terms of modern fracture mechanics concepts. A detailed description of the approach, its merits, and experimental results demonstrating advantages of the method are given in the report on Phase II of this contracted investigation.

# END

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